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Field Evaluation of a Passive Sampling Device For Hydrazines in Ambient Air

P. A. TAFTE,* S. W. BROWN,** A. R. THUROW,***
J. C. TRAVIS****

*GEO-Centers Inc.,
**EG&G, BOC-022, KSC, FL
***Wiltech Corp., KSC, FL
****NASA, KSC, FL

S. L. ROSE-PEHRSSON AND J. R. WYATT

*Chemical Dynamics and Diagnostics Branch
Chemistry Division*

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13. ABSTRACT (Maximum 200 words) A passive sampler-dosimeter has been developed for evaluating part-per billion (ppb) levels of hydrazine and monomethyl hydrazine (MMH) in ambient air. It is designed for quantitative documentation of personnel exposures as well as ambient atmosphere concentrations. It has undergone extensive laboratory and field evaluations. The laboratory tests are reviewed in this report. Tests were conducted in controlled atmospheres to evaluate the following performance parameters: collection rate, face velocity, relative humidity effects, sample stability, reproducibility, linearity, and interference effects of selected chemical vapors. Field tests were conducted to evaluate performance under typical anticipated conditions. The test locations were selected to provide information on the probable interferents. A double-blind protocol was used which involved three groups: industrial hygienists; analytical chemists; and auditors. The data obtained from the field evaluation disclosed a performance problem not encountered in the laboratory. The cause was identified and the prototype system was modified and re-tested. The results from the modified sampler indicate that it is suitable for work place monitoring applications with two minor interferents: tobacco smoke and intense direct sunlight.				
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TABLE OF CONTENTS

INTRODUCTION	1
APPROACH	2
LABORATORY TESTING	5
Test Atmosphere: Generation and Verification	5
Performance Evaluations	6
FIELD TESTING	10
Test Locations	10
Sampling	10
Citrate Badge Analysis	11
Liquid Impinger Analysis	12
Vanillin Color Dosimeter Analysis	13
RESULTS	13
Field Testing	13
Quality Assurance and Quality Control	22
DISCUSSION AND CONCLUSIONS	23
REFERENCES	25
APPENDIX A	27
APPENDIX B	29
APPENDIX C	47
APPENDIX D	69

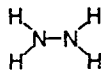
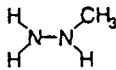
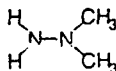
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FIELD EVALUATION OF A PASSIVE SAMPLING DEVICE FOR HYDRAZINES IN AMBIENT AIR

INTRODUCTION

The potential carcinogenicity of hydrazine (Hz), monomethylhydrazine (MMH), and unsymmetrical dimethylhydrazine (UDMH) has caused concern for the health and safety of the workers who may be exposed to them. For brevity the term "hydrazines" in this report is used to mean any of these three hydrazines. The chemical structures of these compounds and their ACGIH [1], NIOSH [2], and proposed [3] recommended exposure limits are listed in Table 1.

Table 1. Recommended Exposure Levels for Hydrazine Propellants

<u>Compound</u>	<u>Structure</u>	<u>ACGIH</u>	<u>NIOSH</u>	<u>Proposed</u>
Hydrazine		0.1 ppm	0.03 ppm	0.01 ppm
Monomethyl Hydrazine		0.2 ppm	0.04 ppm	0.01 ppm
Unsymmetrical Dimethylhydrazine		0.5 ppm	0.06 ppm	0.01 ppm

Monitoring of personnel exposure and the work place environment is necessary to insure that exposure remains below the defined limit and to comply with regulations issued in the Occupational Safety and Health Act of 1970. Two approaches to accomplish this monitoring have been developed by our group--passive sampling and real-time colorimetric dosimetry. Each procedure has advantages and disadvantages. The passive sampler traps and stabilizes the hydrazine for later quantitative analysis; however, it cannot warn the personnel of exposure in real-time. The colorimetric dosimeter provides real-time measurements and can warn personnel of a hazardous condition.

Because the colorimetric dosimeter is directly exposed to the atmosphere its sampling rate depends upon the motion of the air in front of it. A color indicator could be placed behind a diffusion barrier but this would reduce its sensitivity by an order of magnitude making it difficult to read. Thus it is not possible to correlate the exposure of the indicator to the actual concentration in the atmosphere with any certainty. Ideally the colorimetric and the passive systems could be combined onto one badge that could provide an immediate warning and a quantitative record for documentation. Before that can be done each system must be developed independently. This report deals with development of a passive sampler. The real-time dosimeter will be discussed in a separate report.

The reactivity of the hydrazines and their tendency to undergo oxidative decomposition poses a problem to the development of detection systems. A collection scheme is required with the ability to stabilize the hydrazines without interfering with accepted analytical procedures [4].

Current methods of sampling involve detector tubes or midjet impingers with an acidic collection solution. These are "active" sampling methods, meaning they involve the use of a sampling pump to draw the atmosphere through the collection medium. The pumps are bulky and expensive, increasing the size, weight, and cost of the system, placing undesirable constraints upon performance. In addition, a power source is required to operate the pump which places a limit on the sampling period.

Using passive diffusion technology, we have developed a lightweight, inexpensive, sampler that can be used to quantitate ppb exposures to hydrazine and MMH. The following section gives a general description of the sampler and the laboratory tests to characterize its performance. More detailed descriptions are available [5, 6, 7 and 8]. The prototype was evaluated in the laboratory for collection rate, sample stability, reproducibility, linearity, and effects of selected interferents and relative humidity. Following the laboratory characterization, the system was tested at Kennedy Space Center (KSC) in field locations and conditions where it may find future use.

APPROACH

The prototype sampler consists of a coated polyester collection disk and four plastic pieces which included a base, spacer, diffuser, and cap, US Patent 4,780,282. Of these four pieces, the diffuser is the most critical. It controls the collection rate and avoids dependence upon the ambient face velocity. Several design criteria were special for a system design to sample hydrazines. Because of the low exposure limits of hydrazines compared to most other chemicals, it is necessary that the badge sample at a higher rate to obtain sufficient sample for analysis. Hydrazines are polar and reactive precluding the use of metals and most plastics as materials for badge construction. It has been our experience that machined teflon surfaces are unsuitable for sampling low levels of hydrazines.

The most desirable form of a personal sampling device utilizes a passive collection scheme. For an ideal badge design the sampling rate (M) of the passive collector depends only upon the diffusion coefficient (D) of the analyte as described by Fick's first law of diffusion, equation 1.

$$M = D (A/L) (C_1 - C_2) \quad (1)$$

Where: A = The area of the diffusion channel;
L = The length of the diffusion channel;
C₁ = The external (ambient) concentration of the analyte; and
C₂ = The gas-phase concentration of the analyte at the surface of the collector.

Theoretical modeling was employed during the design of a diffuser. Our design is based upon the fact that viscous flow is proportional to A^2/L ; whereas diffusion is proportional to A/L , see

equation 1. Thus increasing the number of holes on the badge, while keeping the total area of the holes constant, decreases the viscous conductance without affecting the diffusion rate. Viscous flow into the badge is due to small pressure differences across the diffuser because of air movement. Additionally, less turbulence is caused at the badge face by many small holes. The disadvantage of increasing the number of holes is the increased surface area of the walls of the holes and greater difficulty in manufacturing the badge face.

Several styles of badge were fabricated and tested. The number and size of holes was varied while maintaining a constant sampling area. Face velocity experiments were performed on the machined badges to select the optimum design. A design having a 2.5 cm diameter pattern of 144 1.0 mm diameter holes was selected for its ability to minimize face velocity effects without severely increasing the detection limit. Designs with fewer, larger diameter holes, exhibited pronounced face velocity effects.

Tests were conducted with badges machined from polyethylene, polypropylene, and teflon. No significant material-dependent differences were found. We were concerned with the potentially detrimental effect of the rough surfaces produced during the drilling of the holes. To minimize this effect, and to aid in the quality control and mass production of the sampler, it was necessary to have the badges molded. Moldsavers, Inc. of Miami, Florida was selected as the manufacturer. Low density polyethylene was the only tested material which could be molded successfully into the desired badge face having the desired hole pattern. The badge was designed to snap together, allowing the cap to be snapped on the back of the base during badge exposure and snapped over the diffuser for storage. The diffuser was designed to snap on the base and to accommodate the cap or a second diffuser. The design of the badge is shown in Figure 1.

The current badge design has 144 one mm diameter holes with a length of 2 mm. Between the diffuser and the substrate there is a 2 mm deep gap 25 mm in diameter. Based upon equation 1 the conductance of the badge is 4.65 cm. This results in a *theoretical* sampling rate of 42, 34, and 29 ml/min for Hz, MMH and UDMH respectively, based upon diffusion constants of 0.154, 0.122, and 0.104 cm²/sec. The measured sampling rate for MMH is 25 ml/min. The theoretical rate may be in error due the assumption that the value of C_2 in equation 1 is zero. By stacking diffusion barriers on top of each other the sampling rate can be decreased. Colorless polyethylene badges were used for initial field tests (K01-K10). Later, black low-density polyethylene badges were used to reduce effects of exposure to strong sunlight, (tests K10A-K18).

The substrate used for the original prototype sampler was a matted polyester drafting film. Initial tests using this material were promising, later it was found to cause the captured MMH on the citric acid to slowly disappear. It is believed that the hydrazine slowly reacts with the substrate. After this discovery, the substrate was changed to Whatman #42 filter paper, which is the substrate currently in use. In laboratory tests, the filter paper substrate did not affect the storage stability of the analyte [7]. Citric acid monohydrate was selected as the coating agent. It has desirable properties as an acid and an antioxidant, additionally it is non-toxic. Using the polyester substrate it was found that the preparation of the citric acid solution was critical to obtaining good results. The solution was made by dissolving citric acid monohydrate in methanol to form a 30% solution. The solution was aged for one week at room temperature and was discarded after two weeks. If retained for longer

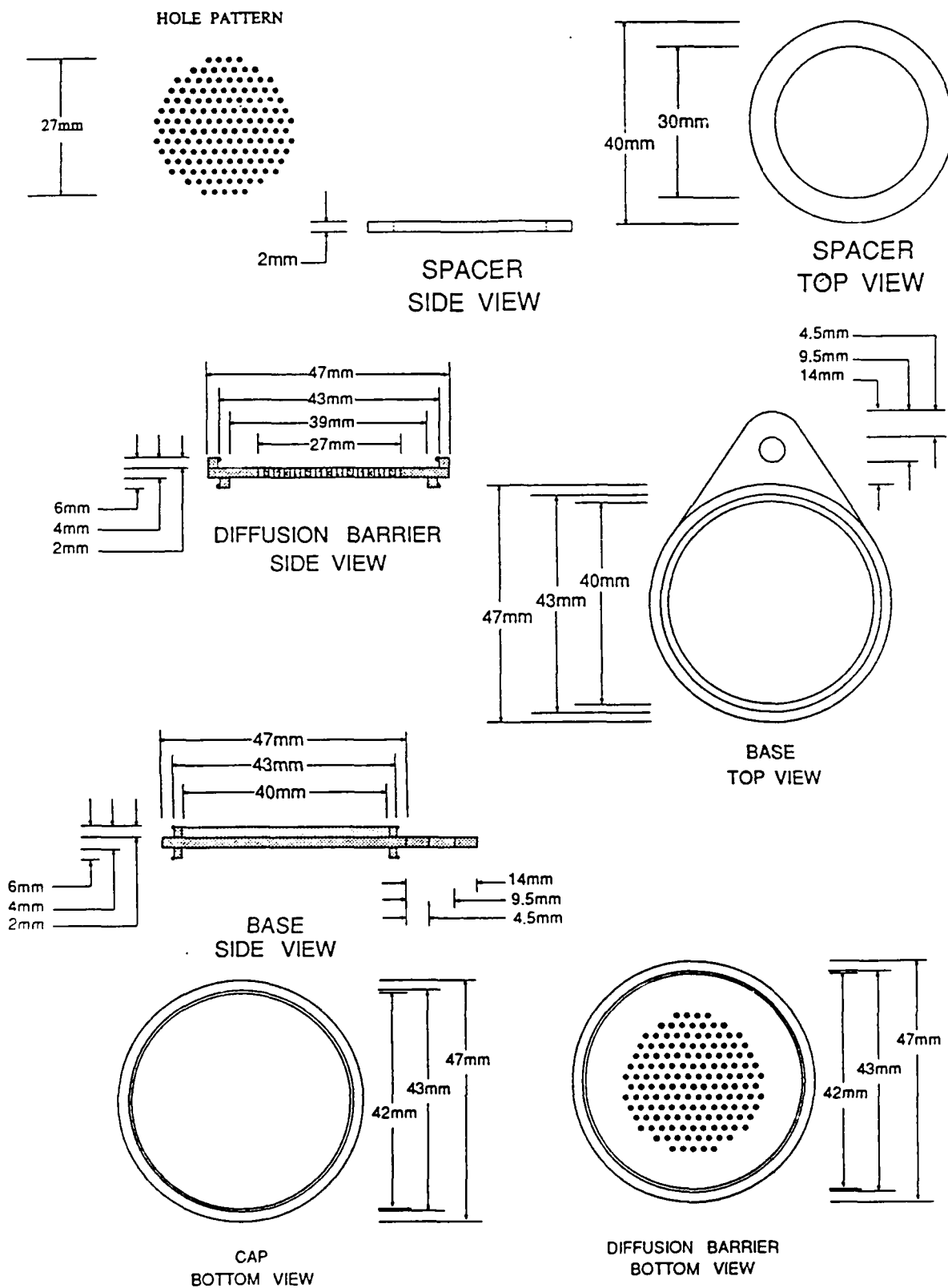


Figure 1. Drawing of the molded polyethylene sampler.

periods, the formation of methyl esters causes significant variations in the coating consistency [5]. Using filler paper substrates, the citric acid solution could be used immediately and stored indefinitely.

The filter paper disks are coated by immersion in the citric acid solution. Teflon-coated tweezers are used to remove the disks. Contact with metals is avoided in order to prevent metal ion catalyzed decomposition of the hydrazines. Large quantities of the coated disks may be prepared and stored in a refrigerator (approximately 3°C) for later use. Prepared samplers have been stored for periods of one month prior to laboratory testing with no effect on performance. Appendix A describes in detail the techniques used for badge preparation.

LABORATORY TESTING

Test Atmosphere: Generation and Verification. The reactivity of the hydrazines makes it necessary to generate dynamically the low ppb levels required for testing. The gas generation system, depicted in Figure 2, can generate hydrazine concentrations from approximately 0.1 to 10 times the TLV (Table 1, ACGIH values) for each compound. Diffusion tubes housed in a constant temperature bath, and continually purged with 100 ml/min of dry nitrogen generate hydrazines. The desired concentration is obtained by adjusting the temperature of the bath, size of the diffusion capillary, and/or the volume of diluent gas.

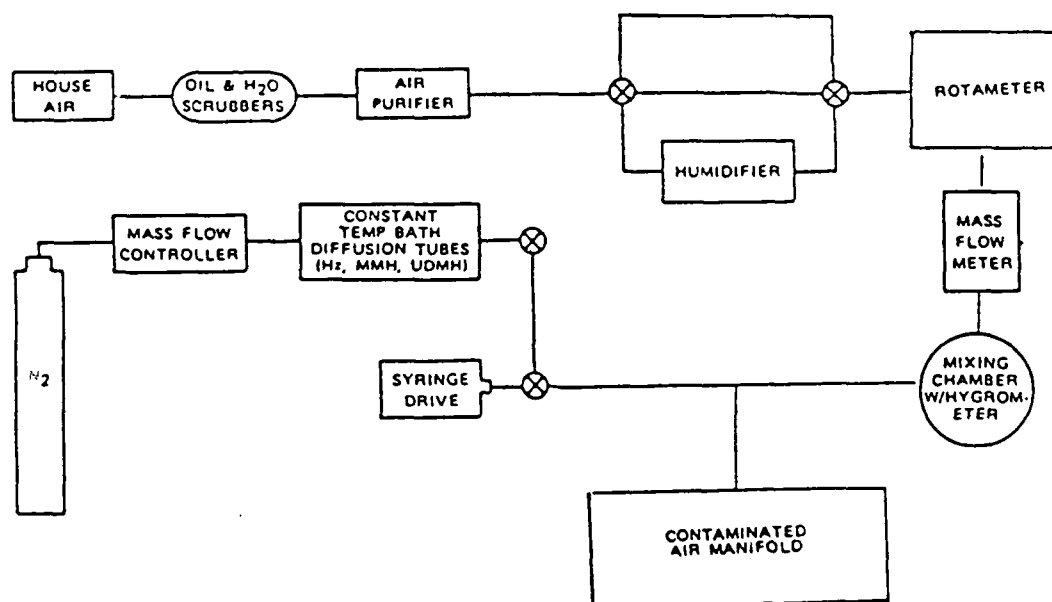


Figure 2. Test gas generator schematic.

Conditioned house-compressed air is used as the diluent. The conditioning procedure consists of passing the house air through a series of demisters, a hot Hopcalite catalyst bed, a reciprocating dual-tower molecular sieve scrubber, and finally through a canister containing potassium permanganate coated alumina (Purafil) and charcoal. The cleaned air is humidified using a stainless steel

gas washer (bubbler) containing distilled, deionized water. Control of the relative humidity is achieved by varying both the gas washer head pressure and the ratio of the humidified to dry air. The moisture content of the air is measured by a hygrometer. Dilution is selected and monitored using calibrated 0 to 10 l/min mass flow controllers.

The exposure experiments were conducted in three similar glass exposure chambers, one of which is depicted in Figure 3. They are cylindrical with conical ends. The exhaust end was removable to allow insertion of the samplers. Teflon baffles were placed at each end to induce laminar flow. The internal diameter of each chamber was different in order to permit the study of a variety of face velocities while holding other gas stream conditions constant. Further variation in face velocity could be attained by varying the flow rate of dilution air in combination with substituting chambers. Table 2 lists the chambers and the conditions available for testing.

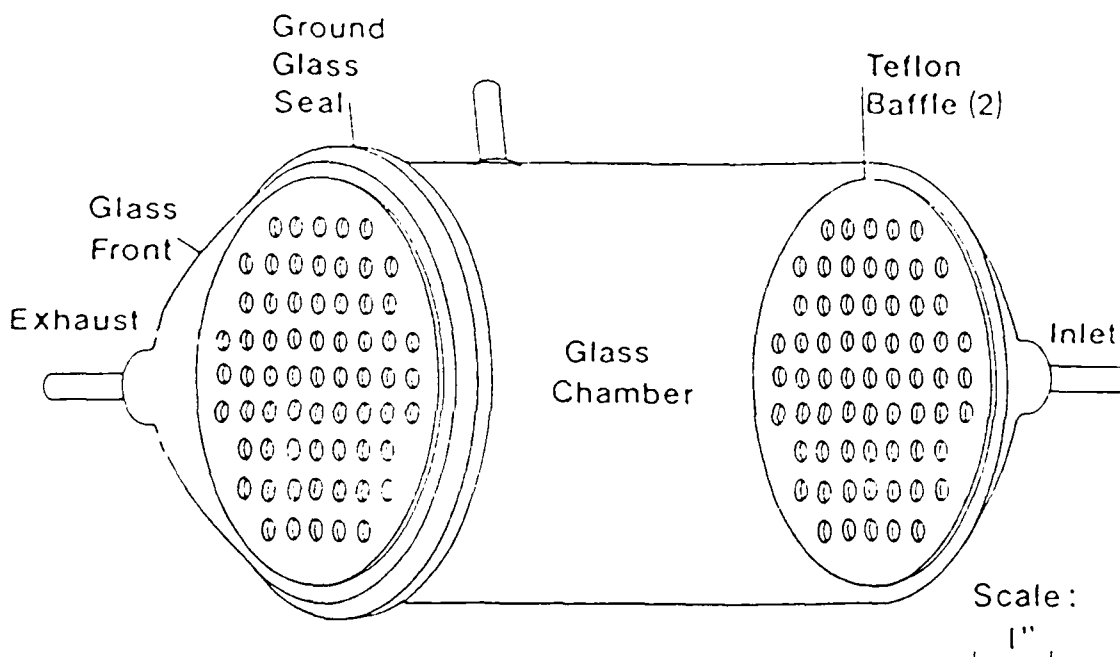


Figure 3. Glass exposure chamber used for laboratory badge testing.

Performance Evaluations. The samplers were prepared as described (Appendix A) and exposed to controlled atmospheres. Typically, four samplers were tested simultaneously. They were placed in the chamber in a 2x2 pattern (each badge in a pair was at the same axial position in the chamber but facing outward). Occasionally six samplers were exposed at one time (2x3 pattern). The badges were mounted on a glass rod suspended between the end baffles of the chamber. This could be done when the concentration and face velocity of the test atmosphere were adequate to prevent depletion of the analyte in the gas stream by the samplers. At low flow rates and concentrations we found that the forward pair of badges captured more hydrazine.

Table 2. Size of the gas exposure chambers and typical conditions.

<u>Diameter</u>	<u>Area</u>	<u>Flow</u>	<u>Face velocity</u>
5.5 cm	23.8 cm ²	5 l/min	335 cm/min* (11 ft/min)
9.0 cm	63.6 cm ²	5 l/min	79 cm/min (2.6 ft/min)
14 cm	154 cm ²	5 l/min	34 cm/min (1.1 ft/min)

* For the small chamber the badge consumed a relatively large portion of the test volume. The face velocity calculations were estimated using the adjusted chamber area.

A variety of equivalent combinations of time and concentration were used to provide conditions for testing the linearity and reproducibility of the sampler. For example, 1 hour at 600 ppb = 3 hours at 200 ppb = 0.6 ppm hours. Exposure times ranged from 0.25 to 65 hours. The concentration of the test atmosphere was verified before and after each exposure experiment by liquid impinger samples that were collected and analyzed using coulometric titration or colorimetric procedures described in Appendix B. In addition, a Thermedics Model 141-1 chemiluminescence instrument and a MDA 7100 paper-tape instrument were occasionally used to monitor the gas stream.

Analysis of the samplers was performed using the coulometric titration procedure described in the analytical portion of the experimental section and detailed in Appendix B. It is not as selective as the colorimetric method, but it is much more sensitive [4]. In laboratory experiments, where no interferences are expected, it is the method of choice.

The effect of face velocity upon the collection rate of the machined prototype diffusers was tested in a MMH gas stream with face velocities of approximately 60, 120, 240, 335, and 670 cm/min (2, 4, 8, 11, and 22 ft/min). The test atmosphere was dry air with 200 ppb MMH. The badges were exposed for five hours. The selected prototype diffuser was tested under the same conditions. The average measured collection rate was 38 ml/min with a minimum of 31 ml/min and a maximum of 45 ml/min [5]. These results are shown in Figure 4.

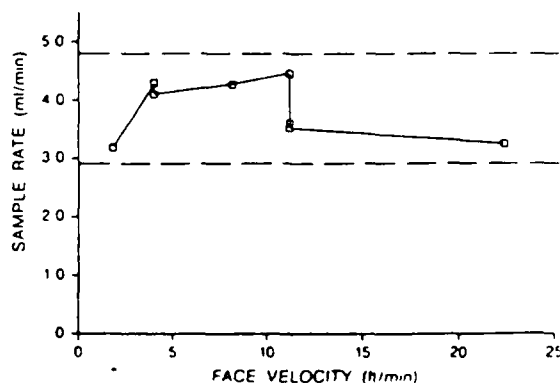


Figure 4. The Effect of Face Velocity on the Sampling Rate of the Machined Prototype Diffuser. The outer lines represent 30% error limits.

The sampling rate of the molded diffuser is approximately 25 ml/min, which is less than that of the drilled prototype. It was calculated from a series of exposures to MMH ranging from 0.25 to 65 hours, Figure 5. Concentrations of MMH between 170 and 500 ppb were used. Each data point represents a test consisting of 4 to 6 individual samples. This and additional data were used to verify the linearity of the sample collection process [5]. The larger sampling rate of the prototype badge is due to the holes being slightly larger than the one millimeter diameter holes in the molded badge.

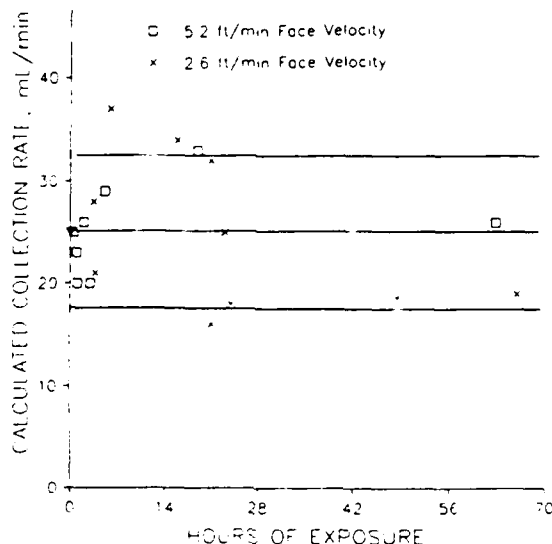


Figure 5. Sampling Rate of the Molded Badge. The center line is the rate, the outer lines are 30% error limits.

The effects of ammonia, freons, and isopropyl alcohol vapors were investigated and no interferences were found [6]. In addition, the collection rate of the dosimeter was found to be independent of the relative humidity of the exposure atmosphere [5].

The stability of the trapped hydrazines was examined by capping and storing exposed prototype badges for periods up to 62 hours. The storage experiments were performed on samples collected from 200 ppb gas streams of MMH at various relative humidities and exposure times. Storage tests were conducted by storing the exposed badges at room temperature and in a refrigerator at 3°C. In addition, the storage of the extracted solution was investigated. Room temperature storage resulted in a significant loss of analyte [5]. A loss of 30% to 75% of the original value was observed after storage for 24 hours. The refrigerated storage or the extraction of the analyte extended the storage stability [8]. This would not allow the badge to be used for long term, low level sampling. For this use it is necessary that the analyte be stable at room temperature.

Initial investigations of the storage instability focused on the citric acid coating. Its composition was investigated by mass spectroscopy and HPLC during the two week aging process [5]. The performance of the solution as a hydrazine trap was also monitored during the same period. Results were inconclusive.

Variations in the substrate material were investigated. Initially, polyester drafting film was used as the substrate. This material wetted well with the citric acid solution, forming a smooth, tacky film. Glass and filter paper materials were tested with the polyester and their performance was compared. The percentage of analyte retained during room temperature storage was greater for glass and filter paper than for polyester. We speculated that the hydrazine reacted with the ester to form a hydrazone which is not easily removed for analysis. Surface microscopy performed by R. Young at NASA KSC indicated that the exposed surface of the polyester was mostly silica and not the ester. The mechanism of analyte loss was not investigated further.

Based on the storage stability data from the substrate study, it was decided to replace the polyester substrate with 4.25 cm diameter disks of Whatman #42 filter paper which is readily available from various chemical supply houses. The disks fit the molded badges, requiring no alterations.

The exposure linearity of the badge was tested by exposure to 200 ppb of MMH for times between 0.25 and 65 hours. The test atmosphere was 45% relative humidity (RH) and a velocity of 79 cm/min (2.2 ft/min). In addition, tests were conducted in which the time was held constant and the concentration was varied between 0.1 and 2 ppm. All of the data except a one hour exposure and a 0.25 hour exposure, fell within the acceptable region, as shown in Figure 6. Fluctuations of the

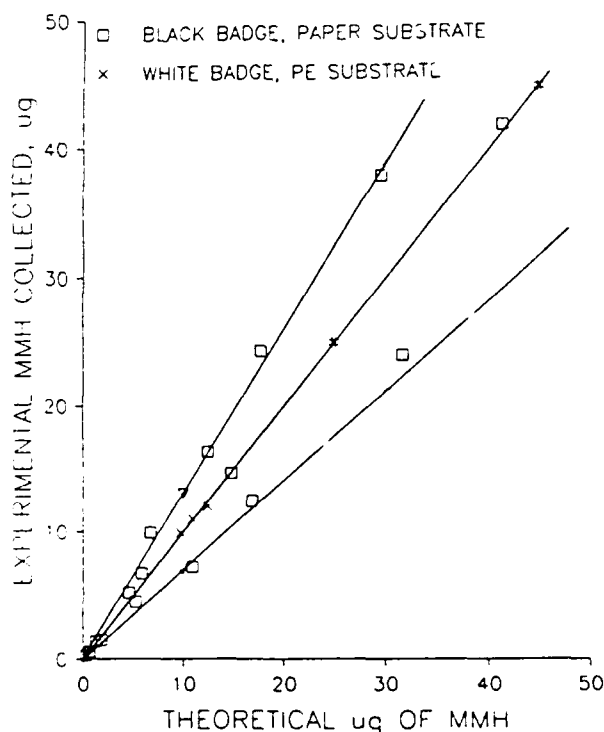


Figure 6. Linearity of the Results Obtained Using the Molded Badge, assuming a collection rate of 25 ml/min. The center line is ideal assuming a 25 ml/min sampling rate, the outer line 30% error limits.

shorter exposures may be due to disruption of the test atmosphere when the badges were placed in the chamber. Adsorption on the badge housing could also be a factor.

FIELD TESTING

Test Locations. Test areas at the Kennedy Space Center were divided into three major categories based on their potential for hydrazine or MMH exposure: unlikely to be exposed, potential exposure, and expected exposure. The locations were selected to encompass the potential field interferences and the effects they may have upon performance. Locations are listed in Table 3.

Sampling. The samplers were prepared by Wiltech Analytical Laboratory at KSC following the procedure described in Appendix A. A group of badges was retained by the analytical laboratory to be used as blanks in their analytical procedures. The blanks were stored in a refrigerator. Samplers for field testing were distributed to the industrial hygienist on the work day preceding the test period.

At each test location, two areas were selected for sampling. A sampling board, holding 12 citrate badges, was placed at each area by EG&G Environmental Health personnel on Monday mornings. The badges were uncapped every morning and recapped at the end of an 8 hour work day. At the end of the sampling period on Monday, Wednesday, and Friday a set of badges was collected for analysis. A set consisted of four badges from each board. Two badges were coded for coulometric analysis (A) and two for verification analysis (B). The exposed badges were submitted to the analytical lab where they were stored in a refrigerator until analysis. In addition, the industrial hygienist submitted a few unexposed badges designated as field blanks. The coulometric analysis was typically performed the first work day following the submission of the sample.

Table 3. Locations Selected for the KSC Field Testing of the Citrate Sampler

<u>Category</u>	<u>Location</u>	<u>Test Number</u>
Unlikely to be Exposed	Hanger S Life Support	K04
	M&O paint shop	K03, K16
	EG&G	K14
	Beach	K10
	Vehicle Assembly Building (VAB) Lounge	K11
Potential Exposure	Hazardous Maintenance Facility (HMF) 96	K02, K18
	Wiltech Labs	K01
	Rotating Service Structure (RSS)	K09, K10A, K15
	Orbiter Processing Facility (OPF)	K13
Expected Exposure	Fuel Storage #1	K06
	Aft Skirt Testing Facility (ASTF)	K07, K08, K12, K17

In addition to the area samples, the citrate badges were also tested as personnel dosimeters. Typically, two workers were monitored; each wearing two citrate badges, an "A" and a "B". These badges were distributed and collected for analysis on a daily basis. Impinger samples (D) were collected daily at the locations of the sample boards in order to verify the exposure the samples received using a validated procedure. Air was drawn through a midjet glass impinger containing 15 ml of 0.1 M H_2SO_4 . Prior to and after sampling, the collection rate of the impinger system was verified to be 200 ml/min using a bubble flow meter. The impingers were submitted to the analytical laboratory where they were stored in the refrigerator until analysis.

In addition to the citrate badge, a colorimetric dosimeter badge was tested. A description of the testing of this indicating system is available [5 and 9]. During the field test, three color badges were placed on each area sampling board on Monday mornings. The dose estimation was evaluated and recorded daily. The disks were collected at the end of the week, stored in zip-lock plastic bags, and sent to NRL for evaluation. On occasion, selected personnel were also monitored with color badges. They were issued a new color badge daily. The used badge was collected, sealed in a zip lock bag and sent with the area samples to NRL for evaluation. Further information on the prototype colorimetric dosimeter will be presented in a future report.

Firebrick samples were scheduled to be used, in place of impingers, in the field tests conducted at White Sands Testing Facility (WSTF). Tabulated results from these tests are available in a report issued by WSTF [9].

The field samples were coded by EG&G using the following label: W - XX - YYY - Z. The key to the label is: W = location, XX = lot #, YYY = sample #, and Z = type of sample. The key for Z is: A = citrate, coulometric analysis; B = citrate, verification analysis (PMA colorimetric or coulometric spike); C = vanillin, D = impinger; and E = firebrick. The analytical laboratory only received the coded samples. The data pertaining to the collection of the samples were recorded by EG&G personnel. The analysis data were recorded by Wiltech. Each group independently sent their data sheets weekly to NRL for compilation. If the analytical laboratory found a quantifiable amount of analyte they would immediately inform the hygienist and the auditor. This was done to allow additional information to be collected by the hygienist while the exposure conditions could be easily recalled.

Citrate Badge Analysis. The coated substrate is removed from the housing assembly with teflon coated tweezers and placed in a glass container. The analyte is desorbed from the disk with a solvent designated by the selected technique. Two accepted wet chemical methods are applicable to this procedure: (1) Coulometric titration miniaturized to achieve the desired sensitivity [11]; and (2) Colorimetric method, phosphomolybdic acid, NIOSH approved method #S149. These methods are detailed in Appendix B, parts 13.3 and 13.2, respectively. The badges were analyzed for MMH exposure unless otherwise specified.

The coulometric titration was used for the laboratory characterization of the badge performance. The schematic of this procedure is shown in Figure 7. It involves the electrochemical generation of bromine from potassium bromide. As the molecular bromine is formed, it instantly reacts with the hydrazine in the solution. When there is no more hydrazine present bromine will accumulate,

forming a redox couple with the bromide. When a redox generated current is measured by the sensing electrode the titration has reached the endpoint. The formula used to calculate the hydrazine in the sample is given in Appendix B. The coulometric procedure is quick, easy and sensitive for analysis of hydrazines, but it is not extremely selective. For the analysis of field samples, the PMA spectrophotometric method was also used. This method is less sensitive, but more selective.

All the "A" badges were analyzed using the coulometric procedure. If a detectable amount of analyte was found, the duplicate "B" badge was analyzed using one of two procedures. The PMA colorimetric analysis was used if the amount detected was greater than the PMA detection limit. If the "A" result was less than the PMA detection limit the coulometric spike procedure was used. In addition, all the Friday "B" badges were analyzed by the PMA method. The unused "B" samples were stored in the refrigerator.

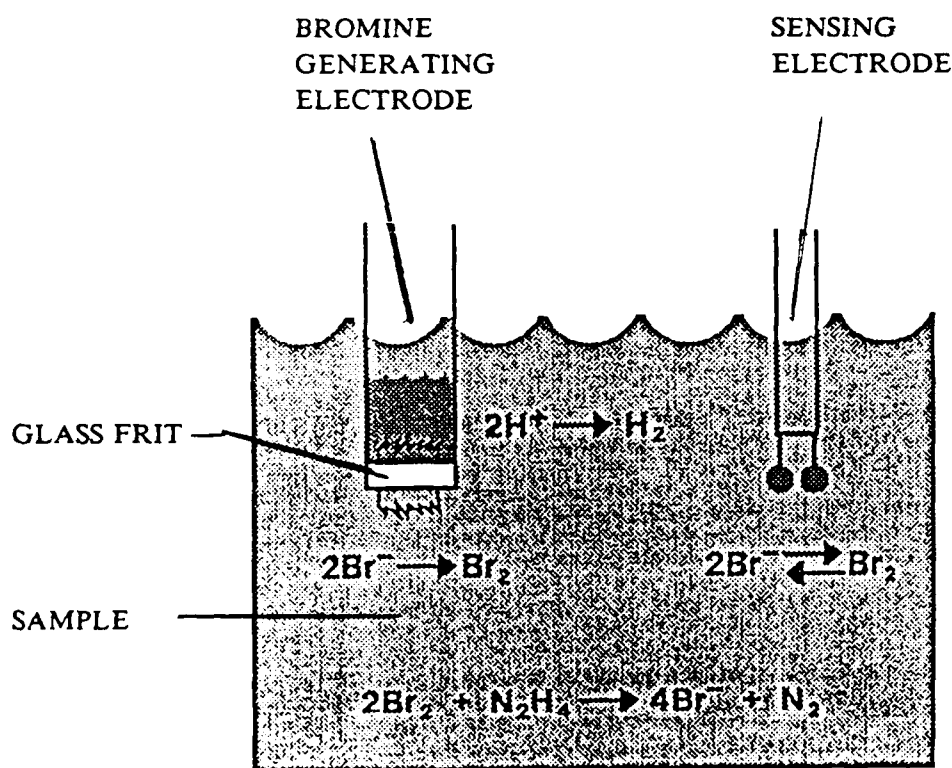


Figure 7. Schematic of coulometric titration.

Liquid Impinger Analysis. The liquid impinger samples, collected to verify the test atmosphere, were analyzed by the ASTM para-dimethylamino benzaldehyde (PDAB) colorimetric method. A copy of the procedure is given in Appendix B (part 8.1 for MMH analysis and 8.2 for Hz analysis). It is based on the condensation reaction of hydrazines with an aldehyde, Figure 8. In the case of unsubstituted hydrazine, two moles of aldehyde can react with one mole of hydrazine to form the azine. The mechanism involves the nucleophilic addition of the nitrogen base, followed by the

elimination of water. This reaction is frequently acid catalyzed by protonation of the carbonyl. The resulting hydrazone absorbs visible light. The ASTM method requires measuring the absorbance spectrophotometrically at 458 nm. These measurements have been shown to conform to Beer's Law, where the amount of absorbed light is proportional to the concentration of the hydrazone in the sample [12].

Vanillin Color Dosimeter Analysis. The same basic chemistry is used with the real-time color badge system. In this case vanillin, 3-methoxy 4-hydroxy benzaldehyde, reacts with the hydrazine. The vanillin is coated on Whatman #42 filter paper and placed in a badge housing that has been modified by cutting away the diffuser section. Hydrazine and MMH present in the ambient environment are trapped on the coated paper where they react with the vanillin indicator; UDMH does not react with the badge to produce a color. The reaction product is detected by the development of a yellow stain on the paper substrate. The intensity of the color is related to the exposure. A color wheel was developed for dose estimation. The dosimeter exposure can be interpolated from a comparison of the badge color with the wheel containing colors equivalent to 0.07, 0.14, 0.48, 1.1, 3.8 ppm-hours of MMH exposure.

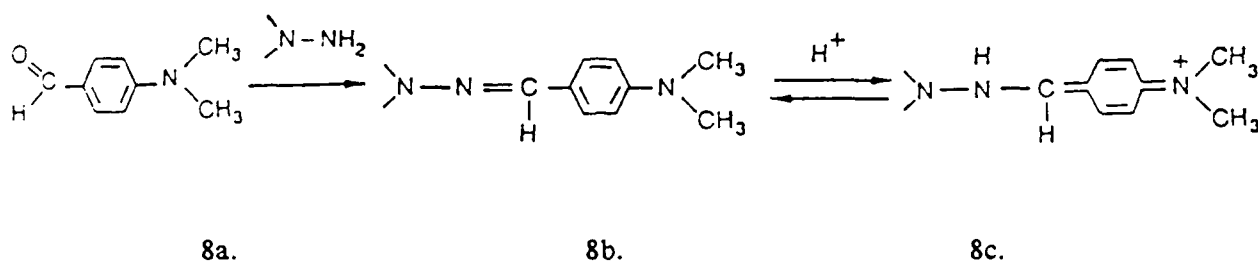


Figure 8. The reaction of PDAB, 8a, with a hydrazine to form the hydrazone, 8b, that becomes yellow on protonation, 8c.

This wheel was used by the industrial hygienists to obtain a dose reading on the field samples (as stated in the sampling procedure). The badges were then sent to NRL. At NRL, the color badges were visually inspected, exposed to MMH and their performance was compared to a control. The control was a sample coated at the same time, but never used in the field.

RESULTS

Field Testing. Eighteen tests were conducted in the field at KSC. Data from test five (K05) was excluded from this report because the samples were left out during a rain storm and were not analyzed. Retesting of the location was performed in test K06. The white badge housing was used for the first tests, K01 through K10. Test K10A was the first field test to use black badges. The black badges were used exclusively for test K10A through K13. In test K14 through K18 white and black badges were tested side-by-side.

A review of the individual tests conducted at KSC is given in Appendix C. The analysis data for each test was tabulated and grouped by the sample type described previously in the report. The industrial hygienists description of the test area is included in Appendix D. The following paragraphs organized by sample type, summarize the results from each test. The data from the field blanks are included in the summaries. The results from the laboratory and EG&G blanks are not in the summaries, but are included in the Appendix C tables.

TEST K01

Location: Wiltech Laboratory

Date: November 1987

Category: Potential Exposure

- A) Two of the twenty-two citrate samples, analyzed by coulometry, indicated analyte present at greater than the detection limit of 0.12 μg . These were not verified by any other method.
- B) The data obtained by the PMA analysis of the citric acid samples are suspected of contamination.
- C) The vanillin colorimetric samples gave no indication of exposure to hypergols.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K02

Location: Hypergol Maintenance Facility M7-961

Date: November 1987

Category: Potential Exposure

- A) Five of the twenty-two citrate samples and one blank, analyzed by coulometric titration, indicated analyte present at greater than the detection limit of 0.12 μg . Two samples were exposed at levels greater than the quantitation limit. All five were personnel samples.
- B) As in test K01, the PMA results for the citrate badges are suspect. The analysis was attempted with and without citric acid added to the standards. The results were still questionable.
- C) No change was noticed on the vanillin color badges.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K03

Location: Maintenance and Operations (M&O) Paint Shop

Date: December 1987

Category: Unlikely to be Exposure

- A) Six of the twenty-two citrate samples analyzed by coulometric titration indicated analyte present at greater than the detection limit of 0.12 μg . Four of these were personnel samples, the other two were five day area samples.

Two of the personnel samples indicated hypergol exposure above the quantitation limit of the coulometric procedure.

- B) The duplicate badges were analyzed by spiked coulometric analysis. The analysis data did not match the "A" data.
- C) No color badges were available for this test.
- D) The daily impingers, analyzed by PDAB, gave no indication of detectable exposure to hypergols.

TEST K04

Location: Hanger S Life Support South Annex

Date: January 1988

Category: Unlikely to be Exposure

- A) One of twenty-two citrate samples analyzed by coulometry indicated a detectable amount of analyte. It was a personnel sample.
- B) None of the duplicate samples was analyzed.
- C) No change was noted on any of the vanillin color badges.
- D) The daily impingers, analyzed by PDAB, gave no indication of detectable exposure to hypergols.

TEST K06

Location: Fuel Storage Area #1

Date: February 1988

Category: Expected Exposure

- A) Eight of the twenty-two citric acid samples analyzed by coulometry indicated a greater than detectable amount of analyte; one was quantifiable.

Area I results were slightly higher than Area II results.

- B) The duplicate samples were analyzed by the coulometric spike procedure. In general the results correlated well with the "A" badge data.
- C) No change was noted on any of the vanillin color badges.
- D) The daily impingers, analyzed by PDAB, gave no indication of detectable exposure to hypergols.

TEST K07

Location: Aft Skirt Test Facility

Date: May 1988

Category: Expected Exposure

- A) All twenty samples analyzed by coulometric titration indicated levels of analyte greater than the quantitation limit of the method, 0.4 μg . Area I samples had significantly greater exposure than Area II samples and appear to increase by $\sim 4 \mu\text{g/day}$. A SCAPE operation was performed on day 3 during the test period, four samples were expected to indicate hypergol exposure.
- B) Five of the twelve samples analyzed by PMA had a detectable amount of analyte. The results did not mimic the coulometric results of "A". The badges were originally analyzed for MMH and the data later corrected for Hz.
- C) Three of the vanillin badges indicated exposure to hypergol. The color was initially noted on only 2 of the 3. Following acidification by HCl vapor the color of the 2 intensified and the third developed color.
- D) The sample collected in Area II on day 3 indicated the presence of hypergol. Again, the analytical lab was not informed that the analyte was Hz so the samples were analyzed as MMH.

TEST K08

Location: Aft Skirt Testing Facility

Date: May 1988

Category: Expected Exposure

- A) All of the citrate samples analyzed by coulometry indicated exposure to hypergols. All but 2 results were greater than the quantitation limit of 0.4 μg . Results from Area I were significantly greater than Area II.
- B) The duplicate citrate samples, analyzed by PMA, did not verify any exposure information.
- C) No change was noted on any of the vanillin color badges.

- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K09

Location: Rotating Service Structure

Date: June 1988

Category: Potential Exposure

- A) All of the citrate samples analyzed by coulometry indicated exposure to hypergols. All the results were greater than the quantitation limit of 0.4 μg . Results from Area I were greater than Area II. Blind blanks also indicated exposures.
- B) The spectrophotometer was broken. The "B" samples were analyzed by coulometric spike procedure. The spiked results correlate well with the corresponding "A" samples.
- C) Four samples indicated a slight exposure to hypergols. All these samples were in Area I with the dose increasing daily.
- D) The samples from day 1 of each area indicate a slight exposure to hypergol.

TEST K10

Location: Beach Location

Date: July 1988

Category: Unlikely to be Exposed

- A) Area samples indicated a high exposure. No hypergols were in this area. The indication must be due to an interferant. The capped blank samples also indicated interference in the coulometric method.
- B) The PMA samples did not verify any MMH exposure. The results were all below detection limit.
- C) One sample had a slight coloration.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K10A

Location: Rotating Service Structure

Date: January 1989

Category: Potential Exposure

- A) This was the first field test conducted using the black badges. Four of the eighteen citrate samples analyzed by coulometric titration gave a response greater than the detection limit. None of the results were equal to or greater than the quantitation limit.

- B) Two of the citrate badges had detectable amounts of analyte by the PMA method. Each of these were 5 day exposure samples from Area II. One was a sample the other was a blank.
- C) No color badges were available for sampling.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K11

Location: Vehicle Assembly Building Break Room

Date: February 1989

Category: Unlikely to be Exposed

- A) Twelve of the eighteen citrate samples analyzed by coulometric titration gave responses greater than the detection limit. Five of the samples indicated quantifiable amounts. First indication of interference due to tobacco smoke.
- B) The samples analyzed by coulometric spike procedure correlated well with the "A" samples. The samples analyzed by PMA did not detect any analyte.
- C) No color badges were available for sampling.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K12

Location: Aft Skirt Testing Facility

Date: February 1989

Category: Expected Exposure

- A) Three of the eighteen citrate badges analyzed by coulometric titration gave results greater than the detection limit. Two samples had quantifiable amounts, these were six day exposures in Area II.
- B) No exposure was verified by PMA analysis of the duplicate citrate badges.
- C) No color badges were available for sampling.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K13

Location: Orbiter Processing Facility

Date: March 1989

Category: Potential Exposure

- A) One sample from the eighteen placed in Areas I and II had a detectable amount of analyte.
- B) The duplicate samples from area I and II did not detect any analyte.
- C) No color badges were available for sampling.
- D) The liquid impinger samples from the second day detected slight exposure.

TEST K13A

Location: EG&G Roof, Horizontal Placement

Date: March 1989

Category: Unlikely to be Exposed

- A) Six of the nine samples had quantifiable amounts of analyte. The samples that did not indicate exposure were 3 capped blanks.
- B) None of the samples were analyzed by an alternate method. The five samples analyzed by the coulometric spike procedure gave similar results to the corresponding "A" samples.
- C) No color badges were available for sampling.
- D) No impinger samples were collected from the area.

TEST K14

Location: EG&G Environmental Health Roof and Remote Antenna Site

Date: May 1989

Category: Unlikely to be Exposed

- A) The citrate samples in black badges indicated quantifiable amounts when placed in both the vertical and horizontal positions. The results from the samples in a horizontal position were much greater than the vertical. The vertical black badges in area 1 and 2 had equivalent results. The black badge blanks did not detect any analyte.

The white badges were only placed in the horizontal position. All the samples gave extremely high results, including the blanks.

- B) The only samples to indicate analyte were the white badge samples placed in a horizontal position.

- C) No color badges were available for sampling.
- D) No impingers collected. No hypergols were anywhere in the vicinity so no verification was needed.

TEST K15

Location: Rotating Service Structure, 39B

Date: August 1989

Category: Potential Exposure

- A) All twelve of the white badges indicated detectable amounts of analyte, nine of the results were above the quantitation limit.

One of the eighteen black badge samples indicated a detectable amount of analyte.
- B) None of the samples analyzed by PMA detected any analyte. The samples analyzed by the coulometric spike procedure gave similar results to corresponding "A" samples.
- C) GMD prototype badges were used for the color dosimeters. They contained two exposure windows, each with a different indicator. The upper window used PDAB and the lower window used Vanillin. The vanillin section did not indicate exposure. The PDAB section developed a slight yellow color; the reaction product formed by exposure to hydrazine is an orange-red.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K16

Location: M&O Paint Shop

Date: August 1989

Category: Unlikely to be Exposed

- A) The white badges consistently gave high results for the coulometric analysis. Four of the six black badges indicated slight exposure with one result greater than the quantitation limit.
- B) Three of the white badge samples analyzed by PMA indicated exposure.
- C) The color badges were not used during this test.
- D) No liquid impinger samples were taken during this test.

TEST K17

Location: Aft Skirt Testing Facility

Date: August 1989

Category: Expected Exposure

- A) The white badges consistently gave high results for the coulometric analysis. Nothing was detected by the black badge samples.
- B) None of the sample analyzed by PMA detected any analyte. The samples analyzed by the coulometric spike procedure gave similar results to corresponding "A" samples.
- C) GMD prototype badges were used for the color dosimeters. They contained two exposure windows, each with a different indicator. The upper window used PDAB and the lower window used Vanillin. The vanillin section did not indicate exposure. The PDAB section developed a slight yellow color; the reaction product formed by exposure to hydrazine is an orange-red.
- D) The daily impingers analyzed by PDAB gave no indication of detectable exposure to hypergols.

TEST K18

Location: Hypergol Maintenance Facility M7-961

Date: August 1989

Category: Potential Exposure

- A) The white badges consistently gave high results for the coulometric analysis with the exception of day one in Area 1. The black badges did not detect any analyte.
- B) None of the sample analyzed by PMA detected any analyte. The samples analyzed by the coulometric spike procedure gave similar results to corresponding "A" samples.
- C) GMD prototype badges were used for the color dosimeters. They contained two exposure windows, each with a different indicator. The upper window used PDAB and the lower window used Vanillin. The vanillin section did not indicate exposure. The PDAB section, of the badges issued for day two, developed a slight yellow color; the reaction product formed by exposure to hydrazine is an orange-red.
- D) No liquid impinger samples were taken during this test.

Details of the results of tests performed at NASA, WSTF are given in separate report [10]. In general, high results were obtained on colorless badge samples exposed to sunlight that were analyzed by coulometry but not with a WSTF ion chromatography method. WTSF found that the black badge provided adequate protection from sunlight exposure. A slight decrease in the concentration of a spiked sample was observed.

Quality Assurance and Quality Control (QA/QC). To verify the proficiency of the analytical laboratory, a set of spiked samples were incorporated into the field test. These badges were prepared at NRL. They were then given to the industrial hygienist for random, blind incorporation. Table 4 details the exposure of the spiked samples. The theoretical loading of the badges was calculated and kept as proprietary information by the auditors.

Table 4. Spiked Citrate Samples for QA/QC

Sample Number	Spiked with	Conc. (ppb)	Duration (hrs)	Volume** (l)	μ g Spiked	μ g Found	Analytical Method
874	MMH	*	4.6	6.9	1.7	1.7	Coul
875	MMH	*	4.6	6.9	2.9	2.5	PMA
880	MMH	*	90.6	136	28	26	Coul
881		*	90.6	136	37	24	PMA
898	MMH	*	16	24	5.6	5.4	Coul
899	MMH	*	16	24	7.4	5.2	PMA
902	MMH	0	0	0	0	0	Coul
903	MMH	0	0	0	0	nd	PMA
1001	MMH	500	67.5	101	95	>87	Coul
1002	MMH	500	67.6	101	95	>27	Coul
1007	MMH	214	16	24	9.7	6.2	PMA
1008	MMH	214	16	24	9.7	11.4	PMA
1009	MMH	214	16	24	9.7	9.0	Coul
1010	MMH	214	16	24	9.7	11.1	Coul
1014	MMH	235	5.5	8.3	3.7	2.3	Coul
1015	MMH	235	5.5	8.3	3.7	4.3	Coul
1016	MMH	235	5.5	8.3	3.7	6.4	PMA
1017	MMH	235	5.5	8.3	3.7	3.9	Coul
1021	Hz	65	5.5	8.3	1.0	1.1	Coul
1022	Hz	65	5.5	8.3	1.0	3.9	PMA
1023	Hz	65	5.5	8.3	1.0	3.9	Coul
1024	Hz	65	5.5	8.3	1.0	1.9	Coul

* Conc. unknown. Amount spiked was determined by analysis of duplicates at NRL.

**Assuming collection rate of 25 ml/min.

The results from the analysis of the spiked samples are listed in Table 4. The analytical methods used were the coulometric titration procedure (Coul) and the PMA colorimetric procedure. The industrial hygienist did not inform the analytical laboratory that some of the samples were to be analyzed for Hz instead of MMH. Because of this, the analytical laboratory analyzed all the samples for MMH exposure. This would not effect the coulometric titration results, but the colorimetric

results could be off for the hydrazine samples. Therefore, the PMA results are of questionable value for sample 1022.

DISCUSSION AND CONCLUSIONS

Laboratory Test. The molded polyethylene badge provides an excellent housing for the collection disk. The diffuser minimizes face velocity effects and establishes a collection rate of 25 ml/minute for MMH. At this sampling rate, detection of MMH at a concentration of 200 ppb MMH requires a ten minute exposure when analyzed by the coulometric titration procedure. An upper detection limit, or saturation limit of the badge, has not been defined. Quantifiable data was obtained from exposures to 200 ppb MMH for 65 hours. Since the badge is simply the collection media, the detection limits of the sampler are dependant upon the analytical method selected. The coulometric procedure is much more sensitive, but less selective than the available colorimetric methods. If better analytical methods were available, then the detection limit could be decreased.

The badge is a simple plastic design and its current production cost is less than \$0.25. Assembly of the badge is simplified by its ability to be securely snapped together. The resulting badge is durable and lightweight. These are desirable qualities for a disposable personal dosimeter. The laboratory performance of the original white badge housing and the black badge modification was acceptable.

The room-temperature instability of the analyte on the original prototype badge was improved by replacing the substrate material used for the collection disk. The original polyester substrate experienced a loss of analyte, decreasing by 30% to 75% in a period of twenty-four hours [5]. The new filter paper substrate has exhibited no significant loss of the analyte for periods of 7 days at room temperature [8]. Long term storage is possible with either system, polyester or paper, if the sample is stored in the refrigerator or extracted and stored as a solution.

The effects of ammonia, freons, and isopropyl alcohol vapors were investigated and no interference effects were found. In addition, the collection rate of the dosimeter was found to be independent of the relative humidity of the exposure atmosphere.

Field Test. The performance of the white badge housing using the filter paper substrate was acceptable for sampling in locations with no sunlight exposure. The sunlight interference was noticed in both coulometric and colorimetric analysis. The effect is much greater when the coulometric analysis is used. Both field tests conducted at KSC and WSTF indicated the sunlight interference effect.

To avoid this interference the badge was modified. The same mold was used to manufacture the sampler, but black polyethylene was used. By substituting the black housing for the white housing it is possible to use the badge in bright sunlight if care is taken not to point the badge directly at the sun for any length of time. The black badge has been field tested and has performed successfully. Field tests, conducted in the intense summer sun at KSC, indicated minimal interference when used in vertical positions, test K14. When used in a horizontal position the sun can penetrate directly through the diffusion holes and interact with the citrate surface, interfering

significantly with the coulometric and colorimetric analysis. As previously mentioned, the effect is greatest with the coulometric procedure. Based on this, we recommend samples that are known to have been exposed to sunlight be analyzed by the colorimetric procedure. Testing has been conducted with the black badge at WSTF. A report containing the results will be issued in the near future. WSTF has informed us that the black badge significantly reduced the sunlight interference effect they had observed with the original white badge.

There was one other interference effect noted during the field testing. Badges placed in the break rooms (lounges), where personnel smoked, exhibited elevated coulometric results, Field Test K11. The tobacco smoke did not interfere with the PMA analysis of the duplicate badges or the PDAB analysis of impingers. We recommend colorimetric analysis for samples that have significant exposure to tobacco smoke.

Personnel found the badge easy to use. Its design allowed it to be worn without interfering with ones duties. The analytical chemists found it simple to prepare and analyze. Application of the badge could be simplified further by use of an identification/data sticker. It would have an assigned sample number and contain spaces for exposure information. Included should be the desired analyte (MMH or Hz) and the preferred analytical technique, if any, based on known exposure to an interferant. When the badge is available for routine use, we feel it will be an asset to the industrial hygienist in documenting Hz and MMH exposures. However, one must remember that passive systems have inherent inaccuracy and results must not be expected to have accuracy greater than 30% of the actual exposure.

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APPENDIX A

Reference 4-0-111 Page 1 of 2 Appendix 13.1

The badge preparation method used by Wiltech Analytical Laboratory are compiled in the following Appendix. The method retains its internal Wiltech reference number under the Appendix title.

13.1 MMH/N₂H₄ DOSIMETER BADGE PREPARATION

13.1.1 Apparatus

- 13.1.1.1 Balance, top load.
- .2 Volumetric flasks, 100 ml.
- .3 Whatman No. 42 filter paper, 4.25 cm
- .4 Forceps, plastic.
- .5 Kimwipes.
- .6 Polypropylene bags, 4 x 4 inches.
- .7 Labels.
- .8 Bag sealer.

13.1.2 Reagents

- 13.1.2.1 Citric acid, monohydrate, HO₂C(OH)C(CH₂CO₂H)₂·H₂O, reagent grade.
- .2 Methanol, CH₃OH, reagent grade.
- .3 D.I. water.

13.1.3 Safety - Refer to Section IV of 4-0-111 for safety requirements and specific hazards, precautions, and emergency procedures concerning fire (Paragraph 4.4.1) and solvents (Paragraph 4.4.3).

13.1.4 Preparation of Coating Solution

- 13.1.4.1 Transfer 30 g of citric acid into a 100 ml volumetric flask.
- .2 Add approximately 80 ml methanol to dissolve all crystals. Add methanol again to mark. Mix.
- .3 Allow the solution to age for at least one full week before using. Solution should be discarded if crystals start to develop in the solution or if the coating applied to the film dries out or crystallizes.

This step is not necessary for badges using paper substrates, see text of this report.

APPENDIX A

Reference 4-0-111 Page 2 of 2 Appendix 13.1

13.1.5 Assembly of Dosimeters

- 13.1.5.1 Wash dosimeter parts with warm soapy water by agitation (do not use brush); rinse with D.I. water; pat dry. Blow the diffuser with GN_2 to ensure no water is lodged in the holes.
- .2 Pour some of the coating solution into a 250 ml beaker and place the filter discs in the solution and allow to soak for 5 minutes.
- .3 Load the disc in the dosimeter holder, ensuring the disc is free of wrinkles or scratches. Press the spacer on top of the disc with forceps.
- .4 Let the coating cure at room temperature with disc uncovered for 3 to 4 minutes.

NOTE

Cured coating should be sticky and shiny, not dried out with crystals.

This step is not necessary for badges using paper substrates, see text of this report.

- .5 Place the diffuser and then the cover on the dosimeter holder.
- .6 Properly label the dosimeter with lab number and data assembled.
- .7 Place the dosimeters in a polypropylene bag and store in a refrigerator.

APPENDIX B

The badge analysis method used by Wiltech Analytical Laboratory are compiled in the following Appendix. The method retains its internal Wiltech reference number under the Appendix title.

APPENDIX B

Reference 4-0-111 Page 1 of 4 Appendix 8.1 PCR-5

8.1 DETERMINATION OF MONOMETHYL HYDRAZINE VAPOR CONCENTRATION IN NITROGEN OR AIR

8.1.1 Apparatus

- 8.1.1.1 Spectrophotometer, UV-VIS, Varian Series 634, or equal.
- .2 Cells, Silica, UV-VIS, 1 cm rectangular.
- .3 Balance, analytical.
- .4 Gas meter, wet test, precision, ASTM D1071, calibrated, or equal.
- .5 Air sampling pump.
- .6 Glass midget impinger, fritted, 170-220 μ maximum pore diameter.
- .7 Pipets, serological, 10 ml.
- .8 Pipets, volumetric, 0.5 ml, 1 ml, 2 ml, 4 ml, 10 ml, 15 ml, and 25 ml.
- .9 Flasks, volumetric, 100 ml, 200 ml, and 500 ml.
- .10 Glass vials with screw caps, 20 ml.
- .11 Graduated cylinder, 250 ml.
- .12 Amber reagent bottle, 250 ml.
- .13 Glass wool.
- .14 Stopcock grease.
- .15 Flow control valve.
- .16 Tubing, Teflon and Tygon, assorted sizes and lengths.

8.1.2 Chemicals

- 8.1.2.1 Sulfuric acid H_2SO_4 , concentrated, reagent grade.
- .2 p-Dimethylaminobenzaldehyde, p-DAB, reagent grade.
- .3 Monomethyl hydrazine sulfate salt, $\text{MMH} \cdot \text{H}_2\text{SO}_4$, reagent grade.
- .4 Methanol, CH_3OH , absolute, reagent grade.

APPENDIX B

Reference 4-0-111 Page 2 of 4 Appendix 8.1 PCR-5

8.1.3 Safety

- 8.1.3.1 General - Refer to Section IV of Toxic Vapor Detector Calibration Manual 4-0-111 for safety requirements and specific hazards, precautions and emergency procedures concerning fire (Paragraph 4.4.1) and hypergols (Paragraph 4.4.4.)
- 8.1.3.2 Safety Equipment (Personal)
 - 8.1.3.2.1 Face shield.
 - .2 Laboratory coat or rubber apron.
 - .3 Gloves, chemical-resistant.
- 8.1.3.3 Safety Equipment (Laboratory)
 - 8.1.3.3.1 Fume hood.
 - .2 Safety shower and eyewash fountain.
 - .3 MMH concentration monitoring device.
 - .4 Fire extinguisher.

WARNING

Monomethyl hydrazine is a suspected carcinogenic chemical. Handle hydrazine only in a fume hood. Avoid all oxidizing agents. Wear personal safety equipment. Note location of the closest fire extinguisher, safety shower, and eyewash fountain. Ensure test area conforms to good housekeeping standards. Monitor working area MMH concentration with a calibrated monitoring device.

8.1.4 Preparation of Reagents

- 8.1.4.1 Sulfuric acid absorbing solution, 0.1 N (nominal)-
Pipet 3 ml of concentrated sulfuric acid into 1 liter volumetric flask containing approximately 500 ml D.I. water, mix, and bring volume to mark with D.I. water.
- .2 p-DAB Solution - Mix 1.6 g p-DAB, 5 ml concentrated H_2SO_4 , and 200 ml methanol in an amber reagent bottle. Store in dark place. Shelf life of the solution is two weeks.

APPENDIX B

Reference 4-0-111 Page 3 of 4 Appendix 8.1 PCR-5

- 8.1.4.3 Monomethyl hydrazine stock solution - 100 ppm - Transfer 0.157 g of MMH-H₂SO₄ salt, weighed to the nearest 0.01 mg, to a 500 ml volumetric flask containing about 100 ml of 0.1 N H₂SO₄. Mix. Fill to the mark with 0.1 N H₂SO₄.
- .4 MMH working standard solutions - Pipet 0.5, 1.0, and 2.0 ml of monomethyl hydrazine stock solution respectively into 100 ml volumetric flasks and bring to volume with 0.1 N H₂SO₄. The concentrations of the working standard solutions are 0.5, 1.0, and 2.0 ppm.
- 8.1.5 Sampling of Monomethyl Hydrazine Vapor
- 8.1.5.1 Set up the sampling apparatus as in Figure 1.
- .2 Pipet 25 ml of 0.1 N H₂SO₄ into the impinger, grease the stopper lightly, and close.
- .3 Turn on the air pump and adjust the flow rate control valve to pass 0.5 liter per minute flow.
- .4 Attach the inlet of midget impinger to hydrazine vapor source with a short piece of Teflon tubing.
- .5 As a guide, sample 6 liters for 1.5 ppm monomethyl hydrazine vapor and 3 liters for 5 ppm monomethyl hydrazine vapor.
- 8.1.6 Analysis of Sample
- 8.1.6.1 Pipet 10 ml of 0.1 N H₂SO₄, 0.5 ppm, 1.0 ppm, and 2 ppm working standard solutions and sample solutions respectively into labeled glass vials. The 10 ml of 0.1 N H₂SO₄ solution is used as reagent blank.
- .2 Pipet 4 ml of p-DAB solution into each vial; cap, and mix thoroughly.
- .3 After 30 minutes, zero the spectrophotometer with reagent blank at 457 nm and slit 2.
- .4 Read the absorbances of the standard and sample solutions against reagent blank.

NOTE

Refer to spectrophotometer instruction manual as required.

APPENDIX B

Reference 4-0-111 Page 4 of 4 Appendix 8.1 PCR-5

- 8.1.6.5 Plot absorbance readings vs. concentrations of the standard solutions.
- .6 Determine from the standard curve the concentrations of MMH present in each sample solution.

8.1.7 Calculation

$$\text{ppm MMH in nitrogen (or air)} = \frac{13.3 A}{V}$$

A = ppm of MMH in sample solution

V = liters of MMH vapor sampled

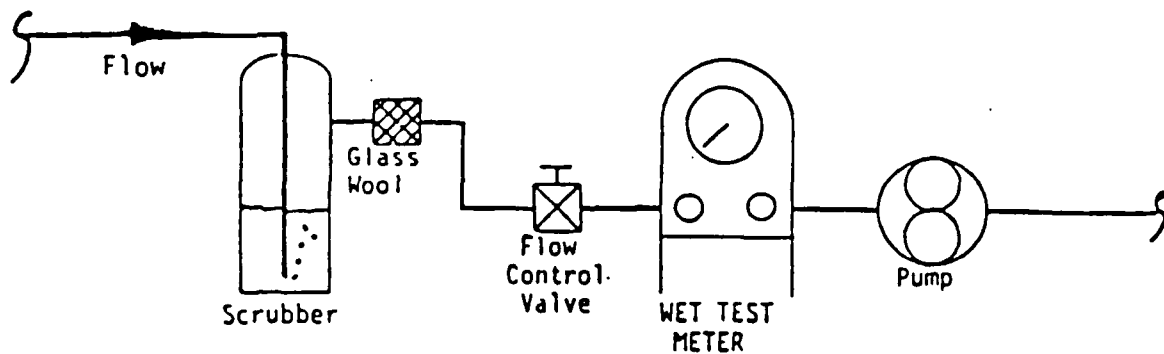


FIGURE 1 - HYPERGOLIC FUEL VAPOR SAMPLING APPARATUS

APPENDIX B

Reference 4-0-111 Page 1 of 4 Appendix 8.2 PCR-6

8.2 DETERMINATION OF HYDRAZINE VAPOR CONCENTRATION IN NITROGEN OR AIR

8.2.1 Apparatus

- 8.2.1.1 Spectrophotometer, UV-VIS, Varian Series 634, or equal.
- .2 Cells, Silica, UV-VIS, 1 cm rectangular.
 - .3 Balance, analytical.
 - .4 Gas meter, wet test, precision, ASTM D1071, calibrated, or equal.
 - .5 Air sampling pump.
 - .6 Glass midget impinger, fritted, 170-220 μ maximum pore diameter.
 - .7 Pipets, serological, 10 ml.
 - .8 Pipets, volumetric, 0.5 ml, 1 ml, 2 ml, 10 ml, 15 ml, and 20 ml.
 - .9 Flasks, volumetric, 100 ml, 200 ml, and 500 ml.
 - .10 Glass vials with screw caps, 20 ml.
 - .11 Graduated cylinder, 200 ml.
 - .12 Amber reagent bottle, 250 ml.
 - .13 Glass wool.
 - .14 Stopcock grease.
 - .15 Flow control valve.
 - .16 Tubing, Teflon and Tygon, assorted sizes and lengths.

8.2.2 Chemicals

- 8.2.2.1 Sulfuric acid, H_2SO_4 , concentrated, reagent grade.
- .2 p-Dimethylaminobenzaldehyde, p-DAB, reagent grade.
 - .3 Hydrazine sulfate, $(N_2H_4) \cdot H_2SO_4$, reagent grade.
 - .4 Methanol, CH_3OH , absolute, reagent grade.

APPENDIX B

Reference 4-0-111 Page 2 of 4 Appendix 8.2 PCR-6

- 8.2.2.5 D. I. water.
- 8.2.3 Safety
 - 8.2.3.1 General - Refer to Section IV of Toxic Vapor Detector Calibration Manual 4-0-111 for safety requirements and specific hazards, precautions and emergency procedures concerning fire (Paragraph 4.4.1) and hypergols (Paragraph 4.4.4.).
 - 8.2.3.2 Safety Equipment (Personal)
 - 8.2.3.2.1 Face shield.
 - .2 Laboratory coat or rubber apron.
 - .3 Gloves, chemical-resistant.
 - 8.2.3.3 Safety Equipment (Laboratory)
 - 8.2.3.3.1 Fume hood.
 - .2 Safety shower and eyewash fountain.
 - .3 N_2H_4 concentration monitoring device.
 - .4 Fire extinguisher.

WARNING

Hydrazine is a suspected carcinogenic chemical. Handle hydrazine only in a fume hood. Avoid all oxidizing agents. Wear personal safety equipment. Note location of the closest fire extinguisher, safety shower, and eyewash fountain. Ensure test area conforms to good house-keeping standards. Monitor working area hydrazine concentration with a calibrated monitoring device.

- 8.2.4 Preparation of Reagents
 - 8.2.4.1 Sulfuric acid absorbing solution, 0.1 N (nominal)-
Pipet 3 ml of concentrated sulfuric acid into 1 liter volumetric flask containing approximately 500 ml D.I. water, mix, and bring volume to mark with D.I. water.
 - .2 p-DAB Solution - Mix 1.6 g p-DAB, 5 ml concentrated H_2SO_4 , and 200 ml methanol in an amber reagent bottle. Store in dark place. Shelf life of the

APPENDIX B

Reference 4-0-111 Page 3 of 4 Appendix 8.2 PCR-6

- 8.2.4.3 Hydrazine stock solution - 100 ppm - Transfer 0.204 g of $(\text{N}_2\text{H}_4)\cdot\text{H}_2\text{SO}_4$ salt, weighed to the nearest 0.01 mg, to a 500 ml volumetric flask containing about 100 ml of 0.1 N H_2SO_4 . Mix. Fill to the mark with 0.1 N H_2SO_4 .
- .4 Hydrazine working standard solutions - Pipet 0.5, 1.0, and 2.0 ml of hydrazine stock solution respectively into 100 ml volumetric flasks and bring to volume with 0.1 N H_2SO_4 . The concentrations of the working standard solutions are 0.5, 1.0, and 2.0 ppm.
- 8.2.5 Sampling of Hydrazine Vapor
- 8.2.5.1 Set up the sampling apparatus as in Figure 1.
- .2 Pipet 25 ml of 0.1 N H_2SO_4 into the impinger, grease the stopper lightly, and close.
- .3 Turn on the air pump and adjust the flow rate control valve to pass 0.5 liter per minute flow.
- .4 Attach the inlet of midjet impinger to hydrazine vapor source with a short piece of Teflon tubing.
- .5 As a guide, sample 6 liters for 1.5 ppm hydrazine vapor and 3 liters for 5 ppm hydrazine vapor.
- 8.2.6 Analysis of Sample
- 8.2.6.1 Pipet 10 ml of 0.1 N H_2SO_4 , 0.5 ppm, 1.0 ppm, and 2 ppm working standard solutions and sample solutions respectively into labeled glass vials. The 10 ml of 0.1 N H_2SO_4 solution is used as reagent blank.
- .2 Pipet 0.5 ml of p-DAB solution into each vial; cap, and mix thoroughly.
- .3 After 30 minutes, zero the spectrophotometer with reagent blank at 457 nm and slit 2.
- .4 Read the absorbances of the standard and sample solutions against reagent blank.

NOTE

Refer to spectrophotometer instruction manual as required.

APPENDIX B

Reference 4-0-111 Page 4 of 4 Appendix 8.2 PCR-6

- 8.2.6.5 Plot absorbance readings vs. concentrations of the standard solutions.
- .6 Determine from the standard curve the concentrations of hydrazine present in each sample solution.

8.2.7 Calculation

$$\text{ppm N}_2\text{H}_4 \text{ in nitrogen (or air)} = \frac{19.1 A}{V}$$

A = ppm of hydrazine in sample solution

V = liters of hydrazine vapor sampled

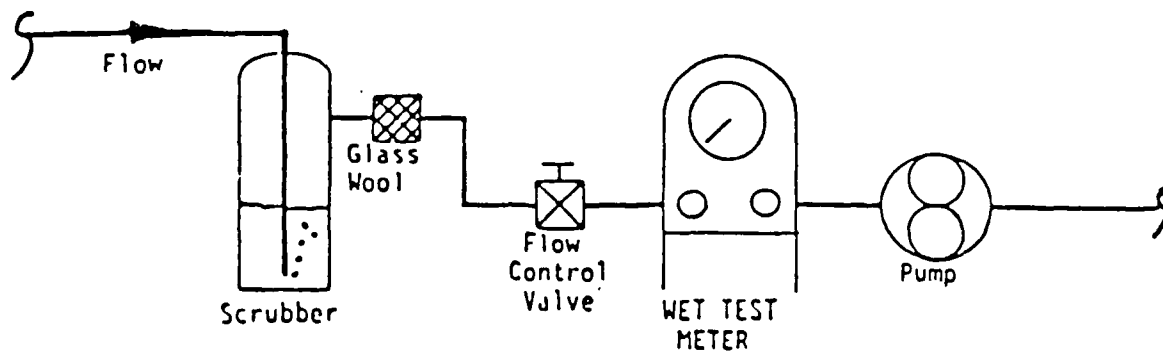


FIGURE 1 - HYPERGOLIC FUEL VAPOR SAMPLING APPARATUS

APPENDIX B

Reference 4-0-111 Page 1 of 4 Appendix 13.2 PCR-11

13.2 DETERMINATION OF MMH AND N_2H_4 CONCENTRATION COLLECTED ON DOSIMETERS USING PHOSPHOMOLYBDIC ACID (PMA) METHOD

13.2.1 Apparatus

- 13.2.1.1 Balance, top load.
- .2 Dark bottle, 500 ml.
- .3 Spectrophotometer, Spectronic 21 or equal.
- .4 Sample tube for spectrophotometer.
- .5 Temperature-controlled environment.
- .6 Volumetric flasks, 25 ml, 100 ml, 2 liter.
- .7 Beakers, 500 ml.
- .8 Graduated cylinder, 500 ml.
- .9 Magnetic stirrer and stirring bar.
- .10 Syringe, 50 μ l.
- .11 Whatman No. 41 filter paper.
- .12 Unexposed dosimeter badges, as prepared in Appendix 13.1.
- .13 Micropipets, 10, 20, 50, and 100 μ l sizes, with disposable tips.

13.2.2 Reagents

- 13.2.2.1 Phosphomolybdic acid, $20MoO_3 \cdot 2H_3PO_4 \cdot 48H_2O$, reagent grade.
- .2 Hydrochloric acid, HCl, reagent grade.
- .3 Hydrazine, N_2H_4 , reagent grade.
- .4 Monomethyl hydrazine, MMH, reagent grade.
- .5 D.I. water.
- .6 Ice.

APPENDIX B

Reference 4-0-111 Page 2 of 4 Appendix 13.2 PCR-11

13.2.3 Safety - Refer to Section IV of 4-0-111 for safety requirements and specific hazards, precautions, and emergency procedures concerning fire (Paragraph 4.4.1), hypergols (Paragraph 4.4.4), and acids (Paragraph 4.4.6).

13.2.3.1 Safety Equipment (Personal)

13.2.3.1.1 Face shield.

.2 Laboratory coat or rubber apron.

.3 Gloves, chemical-resistant.

WARNING

Concentrated sulfuric acid is very corrosive. Wear gloves while handling this chemical.

13.2.3.2 Safety Equipment (Laboratory)

13.2.3.2.1 Fume hood.

.2 Safety shower and eyewash fountain.

.3 MMH and N_2H_4 vapor concentration monitoring devices.

.4 Fire extinguisher.

WARNING

Monomethyl hydrazine and hydrazine are suspected carcinogenic chemicals. Handle MMH and N_2H_4 in a fume hood. Avoid all oxidizing agents. Wear personal safety equipment. Note location of the closest fire extinguisher, safety shower, and eyewash fountain. Ensure test area conforms to good housekeeping standards. Monitor working area MMH and N_2H_4 concentrations with a calibrated monitoring device.

APPENDIX B

Reference 4-0-111 Page 3 of 4 Appendix 13.2 PCR-11

13.2.4 Preparation of Reagents

- 13.2.4.1 Phosphomolybdic acid color develop solution: Stir 9 grams of PMA in 300 ml of D.I. water in a beaker overnight. Filter into a dark bottle and store in a dark place. Do not allow the solid or liquid reagent to contact metal.
- .2 Hydrochloric acid, 0.1N: Add 8.6 ml of HCl to a 2-liter volumetric flask containing approximately 1 liter D.I. water. Add D.I. water to mark and mix.
- .3 Stock hydrazine solution, 320 $\mu\text{g/ml}$: Fill a 100 ml volumetric flask to mark with 0.1N HCl solution. Add, below the surface, 31.7 μl N_2H_4 to the solution. Mix well.
- .4 Stock MMH solution, 320 $\mu\text{g/ml}$: Fill a 100 ml volumetric flask to mark with 0.1N HCl solution. Add, below the surface, 36.6 μl MMH to the solution. Mix well.
- .5 Determine the actual concentration of the stock N_2H_4 or MMH solution by coulometric analysis as outlined in Appendix 8.11 of 4-0-111 using 10 μl of the stock solution for the analysis.
- .6 Reagent blank and working standards: Extract one previously prepared, unexposed dosimeter badge for the reagent blank and each standard to be used. Extract by placing the paper badge in 8 ml of 0.1N HCl and place on a magnetic stirrer for approximately 1 minute. Transfer the solution to a labeled 25 ml volumetric flask. Rinse off badge with 5 ml of 0.1N HCl and add this portion to the contents of the volumetric flask. Add the amount of N_2H_4 or MMH stock solution listed in the table below to each standard flask to yield the desired concentration.

Number of μl	Concentrations	
	N_2H_4	MMH
0 (blank)	0	0
10	3.17	3.20
20	6.34	6.40
50	15.85	16.00
100	31.70	32.00

13.2.5 Analysis of Dosimeter Samples

- 13.2.5.1 Extract the sample badges in the same manner that the unexposed badges were extracted in Paragraph 13.2.4.6.
- .2 Add 7.5 ml of PMA to each of the 25 ml flasks containing blank standards and samples. Fill to mark with 0.1N HCl. Mix.

APPENDIX B

Reference 4-0-111 Page 4 of 4 Appendix 13.2 PCR-11

- 13.2.5.3 Digest each of the 25 ml flasks at 87°C for 50 minutes.
- .4 Cool in ice bath to stop reaction.
- .5 Allow solution to come to room temperature.
- .6 Obtain absorbance readings at 730nm within 30 minutes. Zero instrument with D. I. water. Read reagent blank, standards, and samples against D. I. water.

NOTE

Refer to instrument manual as required.

13.2.6 Calculation

- 13.2.6.1 Plot absorbance vs. concentration of the standards.
- .2 Read the result of the samples directly in μg from the graph.

APPENDIX B

Reference 4-0-111 Page 1 of 4 Appendix 13.3 PCR-12

- 13.3 DETERMINATION OF MMH AND N_2H_4 COLLECTED ON DOSIMETERS USING COULOMETRIC METHOD
- 13.3.1 Apparatus
- 13.3.1.1 Coulometer, 0.1ma, 30mv, with platinum wire electrodes.
- .2 150 ml beaker with 40 ml mark, used as reaction vessel.
- .3 Stirring bar and magnetic stirrer.
- .4 Recorder, Soltec Model 1241, or equal.
- .5 Volumetric flask, 2 liter.
- .6 Forcep, plastic.
- .7 Pipet, graduated, 10 ml.
- .8 Scoop, two scoops yield approximately 0.4 g KBr.
- 13.3.2 Reagents
- 13.3.2.1 Potassium bromide, KBr, reagent grade.
- .2 Sulfuric acid, H_2SO_4 , concentrated, reagent grade.
- .3 D.I. water.
- .4 Hydrazine, N_2H_4 , reagent grade.
- .5 Monomethylhydrazine, reagent grade.
- 13.3.3 Safety
- 13.3.3.1 Refer to Section IV of 4-0-111 for safety requirements and specific hazards, precautions, and emergency procedures concerning fire (Paragraph 4.4.1), hypergols (Paragraph 4.4.4), and acids (Paragraph 4.4.6).
- 13.3.3.2 Safety Equipment (Personal)
- 13.3.3.2.1 Face shield.
- .2 Laboratory coat or rubber apron.
- .3 Gloves, chemical-resistant.

APPENDIX B

Reference 4-0-111 Page 2 of 4 Appendix 13.3 PCR-12

WARNING

Concentrated sulfuric acid is very corrosive. Wear gloves while handling this chemical.

13.3.3.3 Safety Equipment (Laboratory)

13.3.3.3.1 Fume hood.

- .2 Safety shower and eyewash fountain.
- .3 MMH and N_2H_4 vapor concentration monitoring devices.
- .4 Fire extinguisher.

WARNING

Monomethyl hydrazine and hydrazine are suspected carcinogenic chemicals. Handle MMH and N_2H_4 in a fume hood. Avoid all oxidizing agents. Wear personal safety equipment. Note location of the closest fire extinguisher, safety shower, and eyewash fountain. Ensure test area conforms to good housekeeping standards. Monitor working area MMH and N_2H_4 concentrations with a calibrated monitoring device.

13.3.4 Preparation of Reagents

- 13.3.4.1 Sulfuric acid, 0.1M: Pipet 5.6 ml of H_2SO_4 concentration into a 1000 ml volumetric flask containing approximately 700 ml of D.I. water. Add D.I. water to the mark. Mix well.
- .2 Hydrazine stock solution, 100 ppm: Fill a 100 ml volumetric flask to the mark with 0.1M H_2SO_4 solution. Add, below the surface, 10 μ l of N_2H_4 into the solution. Mix well. Solution is stable for one week.
- .3 MMH stock solution, 100 ppm: Fill a 100 ml volumetric flask to the mark with 0.1M H_2SO_4 solution. Add, below the surface, 11.4 μ l of MMH into the solution. Mix well. Solution is stable for one week.

APPENDIX B

Reference 4-0-111 Page 3 of 4 Appendix 13.3 PCR-12

13.3.5 Instrument Preparation

13.3.5.1 Set recorder parameters as follows:

Range - 500 mv
Speed - 60 cm per hour

- .2 Turn coulometer power on.
- .3 Fill the glass vessel containing a stirring bar to the mark with 0.1M H_2SO_4 .
- .4 Place the glass vessel on the magnetic stirrer. Stir at medium speed.
- .5 Add 1.5 scoops of KBr to the solution. Stir until KBr is dissolved.
- .6 Place the electrodes in the solution. Ensure the electrolyte (0.1M H_2SO_4) in the bromine generating electrode is approximately 1 inch above the solution in the vessel.
- .7 Turn on the recorder and lower the pen to start recording. When the line on the chart paper is level, simultaneously activate the coulometer CELL switch and the recorder marker.
- .8 When the recording line deflects upward approximately 1-1/2 inches, deactivate the cell switch, lift up the recorder pen, and promptly place the electrode in clean D.I. water.
- .9 Discard the solution in the vessel; rinse the vessel with D.I. water, then with 0.1M H_2SO_4 .
- .10 Measure the reaction time in terms of distance in cm.
- .11 Repeat Paragraphs 13.3.6.3 and 13.3.6.10 until three consecutive runs indicate the same distance. This is the blank value.
- .12 To ensure that the instrument is functioning properly, repeat Paragraphs 13.3.6.3 to 13.3.6.10 with 30 μ l of N_2H_4 stock solution added to 0.1M H_2SO_4 in the vessel. The measured distance should be 6.0 cm \pm 0.2 cm. If this distance cannot be achieved, notify the shift chemist.

APPENDIX B

Reference 4-0-111 Page 4 of 4 Appendix 13.3 PCR-12

13.3.6 Analysis of Dosimeter Samples

- 13.3.6.1 Place dosimeter spacer and disc into the reaction vessel containing approximately 35 ml 0.1M H₂SO₄ and stir for approximately 30 seconds.
- .2 While taking them out, rinse the spacer and disc with approximately 10 ml 0.1M H₂SO₄. Ensure the volume of solution in the reaction vessel is 40 ml.
- .3 Repeat Paragraphs 13.3.6.4 through 13.3.6.10.

13.3.7 Calculation

moles MMH (or N₂H₄) =

$$\frac{(\text{cm specimen} - \text{cm blank}) \times 60 \frac{\text{sec}}{\text{min}} \times (.1 \times 10^{-3} \text{ amp})}{\text{chart speed } \frac{\text{cm}}{\text{min}} \times 96486 \times 4e^{-} \frac{(\text{amp-sec})}{\text{mole}}}$$

$$\mu\text{g MMH} = \text{moles MMH} \times 4.6 \times 10^7$$

$$\mu\text{g MMH} = \text{net cm for analysis titration} \times 0.715$$

$$\mu\text{g N}_2\text{H}_4 = \text{moles N}_2\text{H}_4 \times 3.2 \times 10^7$$

$$\mu\text{g N}_2\text{H}_4 = \text{net cm for analysis titration} \times 0.497$$

$$\text{PPM MMH in Air} = \frac{(\mu\text{g MMH}) (24.45 \text{ l/mole})}{(46.07 \text{ g/mole (l sampled)})}$$

$$\text{PPM N}_2\text{H}_4 \text{ in Air} = \frac{(\mu\text{g N}_2\text{H}_4) (24.45 \text{ l/mole})}{(32.05 \text{ g/mole (l sampled)})}$$

APPENDIX C

Data tables for the KSC Field Testing of the Citrate Sampler.

The data for each individual test is presented in one table.

The first column indicates the type of sample and distinguishes area and personnel samples.

The second column is used to designate the sampling site. At each test location two sites were selected.

The column titled "Day" indicates the day(s) the sample was exposed. For samples that were placed on a Monday and collected on Friday a "to 5" would appear.

The results of the analysis are in the remaining columns labeled A, B, C, and D.

The "A" column contains the coulometric titration results.

The "B" column contains the PMA results unless indicated by an * which indicates the coulometric spike procedure was used.

The "C" column is used for color badge results. NC is used to indicate No color.

The "D" column is used for the PDAB results obtained from the liquid impinger samples.

DATA FROM FIELD TEST K01

TYPE	AREA	DAY	MICROGRAMS		PPM	PPM
			A	B	HRS C	D
CITRATE PERSONNEL	1	1	0			
	2	1	0.11			
	1	2	0.07			
	2	2	0.07			
	1	3	0.11			
	2	3	0.07			
	1	4	0.14			
	2	4	0.07			
	1	5	0.07	3.5		
	2	5	0.07	2.92		
CITRATE AREA	1	TO 1	0.04			
	1	TO 1	0.07			
	2	TO 1		*0.07		
	2	TO 1	0.04			
	1	TO 3	0.07			
	1	TO 3	0.11			
	2	TO 3	0.07			
	2	TO 3	0.18			
	1	TO 5	0.07	3.11		
	1	TO 5	0.07	2.92		
	2	TO 5	0.07	1.96		
	2	TO 5	0.04	2.53		
	CITRATE LAB BLANK	WT		0.04		
WT			<0.04			
WT			0.04			
WT			0.07			
COLOR AREA	1	TO 5			NC	
	2	TO 5			NC	
IMPINGER AREA	1	1				<0.05
	2	1				0.06
	1	2				<0.05
	2	2				<0.05
	1	3				<0.05
	2	3				<0.05
	1	4				<0.05
	2	4				<0.05
	1	5				<0.05
	2	5				<0.05

* Analyzed by coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K02

TYPE	AREA	DAY	MICROGRAMS		PPM	
			A	B	HRS C	PPM D
CITRATE PERSONNEL BOTH		1	0.07	3.7		
		1	0.04			
		2	0.21	4.2		
		2	0.79	3.8		
		3	0.32	3.1		
		3	0.25	2.8		
		4	0.07	*0.34		
		4	0.47	*0.18		
		5	0.04	3.6		
		5	0.11	2.5		
CITRATE AREA	1	TO 1	0.04			
	1	TO 1	0.07			
	2	TO 1	0.11			
	2	TO 1	0.07			
	1	TO 3	0.04			
	1	TO 3	0.04			
	2	TO 3	0.04			
	2	TO 3	0.04			
	1	TO 5	0.04	2.1		
	1	TO 5	<0.04	2.1		
	2	TO 5	<0.04	2.1		
	2	TO 5	<0.04	2.5		
CITRATE FIELD BLANK			0.07			
			0.11			
			0.36			
			0.11			
CITRATE LAB BLANK WT				3		
			0.07			
			<0.04			
			<0.04			
			0.04			
			0.07			
			0.07			
COLOR AREA	(1)	TO 5			NC	
	(2)	TO 5			NC	
IMPINGER AREA	1	1				<0.05
	2	1				<0.05
	1	2				<0.05
	2	2				<0.05
	1	3				<0.05
	2	3				<0.05
	1	4				<0.05
	2	4				<0.05
	1	5				<0.05
	2	5				<0.05

* Analyzed by the coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant Limit = 0.4 ug (coul)

DATA FROM FIELD TEST K03

TYPE	AREA	DAY	MICROGRAMS		PPM	
			A	B	HRS C	PPM D
CITRATE PERSONNEL	1	1	1.36	*0.14		
	2	1	0.07			
	1	2	0.04			
	2	2	0.07			
	1	3	0.11			
	2	3	2.6	*0.86		
	1	4	0.07			
	2	4	0.14			
	1	5	0.11			
	2	5	0.18			
CITRATE AREA	1	TO 1	<0.04			
	1	TO 1	<0.04			
	2	TO 1	0.04			
	2	TO 1	0.04			
	1	TO 3	0.07			
	1	TO 3	0.07			
	2	TO 3	0.07			
	2	TO 3	0.07			
	1	TO 5	0.07			
	1	TO 5	0.25			
	2	TO 5	0.11			
	2	TO 5	0.14			
CITRATE FIELD BLANK			0.07			
			0.11			
			0.04			
CITRATE LAB BLANK	WT		0.07			
	WT		<0.04			
	WT		0.04			
CITRATE BLANK			0.1			
COLOR					N/A	
IMPINGER AREA	1					<0.05
	2					<0.05
	1					<0.05
	2					<0.05
	1					<0.05
	2					<0.05
	1					<0.05
	2					<0.05
	1					<0.05
	2					<0.05

* Analyzed by coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K04

TYPE	AREA	DAY	MICROGRAMS		PPM	PPM
			A	B	HRS C	D
CITRATE PERSONNEL	BOTH	1	0.14			
	BOTH	1	0.04			
	BOTH	2	0.07			
	BOTH	2	0.04			
	BOTH	3	0.04			
	BOTH	3	0.11			
	BOTH	4	0.04			
	BOTH	4	0.04			
	BOTH	5	0.04			
	BOTH	5	0.04			
CITRATE AREA	1	TO	1	0.04		
	1	TO	1	0.04		
	2	TO	1	0.04		
	2	TO	1	0.04		
	1	TO	3	0.07		
	1	TO	3	0.04		
	2	TO	3	0.04		
	2	TO	3	0.04		
	1	TO	5	<0.04		
	1	TO	5	0.04		
	2	TO	5	<0.04		
	2	TO	5	<0.04		
CITRATE FIELD BLANK	1			<0.04		
	2			<0.04		
	EG&G			0.04		
	EG&G			0.0		
CITRATE LAB BLANK	WT			0.04		
	WT			0.04		
	WT			0.04		
	WT			0.04		
CITRATE BLANK				0.07		
COLOR AREA	1	TO	5		NC	
	2	TO	5		NC	
COLOR PERSONNEL	BOTH	TO	5		NC	
	BOTH	TO	5		NC	
	BOTH	TO	5		NC	
	BOTH	TO	5		NC	
IMPINGER AREA	1	1				<0.05
	2	1				<0.05
	1	2				<0.05
	2	2				<0.05
	1	3				<0.05
	2	3				<0.05
	1	4				<0.05
	2	4				<0.05
	1	5				<0.05
	2	5				<0.05

* Analyzed by coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant Limit = 0.4 ug (coul)

DATA FROM FIELD TEST K06

TYPE	AREA	DAY	MICROGRAMS		PPM	
			A	B	HRS C	PPM D
CITRATE PERSONNEL		2	0.04			
		2	0.21	*0.57		
		4	0.04			
		4	0.04			
CITRATE AREA	1	TO 1	0.32	*0.21		
	1	TO 1	0.36	*0.32		
	2	TO 1	0.07			
	2	TO 1	0.11			
	1	TO 3	0.32			
	1	TO 3	0.18	*0.38		
	2	TO 3	0.07	*0.31		
	2	TO 3	0.18			
	1	TO 4	0.07			
	1	TO 4		*0.21		
	2	TO 4	0.14	*0.5		
	2	TO 4	0.13	*0.54		
CITRATE BLANK	EG&G	2	0.02			
	EG&G	2	0.04	*4		
	EG&G	4	0.79	*1		
	EG&G	4	0.07			
	EG&G	4	VOID	*0.14		
	EG&G	4	0.04	*0.04		
	EG&G	TO 4	0.04	*0.04		
CITRATE FIELD BLANK	1	TO 2	0.07	*0.45		
	2	TO 2	0.61	*0.04		
	1	TO 3	0.04			
	2	TO 4	0.04	*0.04		
CITRATE LAB BLANK	WT		0.04			
	WT		0.04			
	WT		0.01			
COLOR AREA	1	TO 1			NC	
	2	TO 1			NC	
	1	TO 2			NC	
	2	TO 2			NC	
	1	TO 3			NC	
	2	TO 3			NC	
	1	TO 4			NC	
	1	TO 4	**0.11		NC	** Suspected of misnumbering
	2	TO 4			NC	
	2	TO 4			NC	
IMPINGER AREA	1	1			<0.05	
	2	1			<0.05	
	1	2			<0.05	
	2	2			<0.05	
	1	3			<0.05	
	2	3			<0.05	
	1	4			<0.05	
	2	4			<0.05	

*Analyzed by coulometric spike procedure. Detection Limit = 0.12 ug (coul) Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K07

TYPE	AREA	DAY	MMH MICROGRAMS		PPM		COMMENT
			A	B	HRS C	PPM D	
CITRATE	AREA	1	TO	1	3.18	<0.5	
		1	TO	1	4.58	0.9	
		2	TO	1	1.57	<0.5	
		2	TO	1	0.57	0.7	
		1	TO	3	8.65	2.2	
		1	TO	3	18.66	1.1	
		2	TO	3	3.75	<0.5	
		2	TO	3	1.86	<0.5	
		1	TO	5	13.59	0.7	
		1	TO	5	23.67	<0.5	
		2	TO	5	2.71	<0.5	
		2	TO	5	2.57	<0.5	
		CITRATE	PERSONNEL	4			5.58
4					5.11	4.29	
5					8.79	<0.5	
5					11.26	2.6	
CITRATE	BLANK	1			8.4		
		1			12.1		
		2			1.75		
		2			1.43		
COLOR	AREA	1	TO	1			NC
		1	TO	2			NC
		1	TO	3			NC
		1	TO	4			NC
		1	TO	5			NC
		2	TO	1			NC
		2	TO	2			NC
		2	TO	3			NC
		2	TO	4			COLOR AFTER AFTER HCL EXPOSURE
		2	TO	5			<0.07 " "
IMPINGER	AREA	1					<0.05
		2					<0.05
		1		2			<0.05
		2		2			<0.05
		1		3			<0.05
		2		3			0.28
		1		4			<0.05
		2		4			<0.05
		1		5			<0.05

Detection Limit = 0.12 ug (coul)
Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K08

TYPE	AREA	DAY	HYDRAZINE MICROGRAMS		PPH		COMMENT
			A	B	HRS C	PPH D	
CITRATE AREA	1	TO 1	5.29	<1.5			
	1	TO 1	6.21	<1.5			
	1	TO 3	4.85	<1.5			
	1	TO 3	5.96	<1.5			
	1	TO 5	9.02	<1.5			
	1	TO 5	9.99	<1.5			
	2	TO 1	1.42	<1.5			
	2	TO 1	0.97				
	2	TO 3	1.12	<1.5			
	2	TO 3	0.75	<1.5			
	2	TO 5	0.37	<1.5			
	2	TO 5	0.12	<1.5			
	CITRATE BLANKS	1	TO 1	3.85			
1		TO 2	3.16				BLIND BLANKS
1		TO 3	3.21				"
1		TO 4	5.09				"
1		TO 5	4.35				"
2		TO 1	2.24				"
2		TO 2	0.55				"
2		TO 3	1.09				"
2		TO 4	1.64				"
CITRATE BLANK	OFFICE		<0.03				
	OFFICE		0.22	<1.5			
COLOR AREA	1	TO 1			NC		
	1	TO 2			NC		
	1	TO 3			NC		
	1	TO 4			NC		
	1	TO 5			NC		
	2	TO 1			NC		
	2	TO 2			NC		
	2	TO 3			NC		
	2	TO 5			NC		
IMPINGER AREA	1	1				<0.02	
	2	1				<0.02	
	1	2				<0.02	
	2	2				<0.02	
	1	3				<0.02	
	2	3				<0.02	
	1	4				<0.02	
	2	4				<0.02	
	1	5				<0.02	
	2	5				<0.02	

Detection Limit = 0.12 ug (coul)
Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K09

TYPE	AREA	DAY	MICROGRAMS		PPM		COMMENTS
			A	B	HRS C	PPH D	
CITRATE AREA	1	TO 1	5.08	--			
	1	TO 1	5.61	N/A			
	1	TO 3	6.36	--			
	1	TO 3	8.58	--			
	1	TO 3	7.61	--			
	1	TO 3	8.08	* 7.6			
	2	TO 1	3.00	*2.99			
	2	TO 1	2.54	*2.56			
	2	TO 3	5.68	--			
	2	TO 3	2.72	N/A			
	2	TO 3	3.54	*3.52			
	2	TO 3	3.25	N/A			
CITRATE BLANK	1	TO 1	3.47				
	1	TO 2	2.93				
	1	TO 3	4.25				
	1	TO 3	2.90				
	1	TO 3	3.18				
	2	TO 1	2.11				
	2	TO 2	1.93				
	2	TO 3	2.25				
	2	TO 3	1.75				
	2	TO 3	>43.8				SUSPECT DATA
COLOR AREA	1	TO 1			<0.07		
	1	TO 2			0.07		
	1	TO 3			0.14		
	1	TO 3			0.14		
	1	TO 3			0.14		
	2	TO 1			<0.07		
	2	TO 2			<0.07		
	2	TO 3			<0.07		
	2	TO 3			<0.07		
	2	TO 3			<0.07		
IMPINGER AREA	1	1				0.05	
	2	1				0.1	
	1	2				<0.1	
	2	2				<0.1	
	1	3				<0.1	
	2	3				<0.1	

* Analyzed by coulometric spike procedure.

-- SPEC 20 broken, samples lost.

Detection Limit = 0.12 ug (coul)

Quant Limit = 0.4 ug (coul)

DATA FROM FIELD TEST K10

TYPE	AREA	DAY	MICROGRAMS	PPM		COMMENTS
				A	B	
CITRATE	AREA	1	TO 1	12.58	<1.5	
		1	TO 1	13.01	<1.5	
		1	TO 3	27.78	<1.5	
		1	TO 3	20.20	<1.5	
		1	TO 5	--	<1.5	WET
		1	TO 5	--	<1.5	"
* CITRATE	AREA	1	TO 1	0.04		
		1	TO 1	0.07		
		1	TO 3	2.07		
		1	TO 3	1.5		
		1	TO 5	--		
		1	TO 5	--		
CITRATE	BLANK	2	3	2.82		
		2	4	1.32		
		2	5	1.72		
CITRATE	BLANK	1	TO 1	6.44	<1.5	
		1	TO 2	12.19	<1.5	
		1	TO 3	6.47	<1.5	
		1	TO 4	8.72	<1.5	MAY HAVE BEEN WET, DRY AT ANALYSIS TIME
		1	TO 5	13.94	<1.5	WET
COLOR	AREA	1	TO 1		NC	
		1	TO 2		NC	
		1	TO 3		<0.07	
		1	TO 4		--	WET
		1	TO 5		--	CONTAMINATED WITH SAND
IMPINGER	AREA	1	1		<0.05	
		1	1		<0.05	
		1	2		<0.05	
		1	2		<0.05	
		1	3		<0.05	
		1	3		<0.05	
		1	4		<0.05	
		1	4		<0.05	
		1	5		<0.05	

BLANKS AND STANDARDS

	7/12/88	7/13/88	7/14/88	7/15/88	7/18/88
100 PPM STD	96.5	104	106	106	105
WT BLK 1	0.11	0.11	0.18	.57	.18
WT BLK 2	0.18	0.11	0.18	1.07, .43	1.07
MYLAR BLK	0.07		0.07	<.04	.14

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

* A Mylar substrate was used for these samples.

Detection Limit = 0.12 ug (coul)

DATA FROM FIELD TEST K10A

TYPE	AREA	DAY	MICROGRAMS		PPM		COMMENTS
			A	B	HRS	PPM	
					C	D	
CITRATE AREA	1	TO 1	--	<1.0			
	1	TO 1	<0.04	<1.0			
	1	TO 3	<0.04	--			
	1	TO 3	<0.04	<1.0			
	1	TO 5	<0.04	--			
	1	TO 5	0.09	<1.0			
	2	TO 1	0.04	<1.0			
	2	TO 1	0.21	<1.0			
	2	TO 3	0.09	<1.0			
	2	TO 3	0.27	<1.0			
	2	TO 5	0.13	<1.0			
	2	TO 5	0.09	1.0			
CITRATE BLANK	1	TO 1	<0.04	<1.0			
	1	TO 3	<0.04	<1.0			
	1	TO 5	0.13	<1.0			
	2	TO 1	<0.04	<1.0			
	2	TO 3	<0.04	<1.0			
	2	TO 5	0.09	1.2			
IMPINGER AREA	1	1					<0.05
	2	1					<0.05
	1	2					<0.05
	2	2					<0.05
	1	3					<0.05
	2	3					<0.05
	1	4					<0.05
	2	4					<0.05
	1	5					<0.05
	2	5					<0.05

BLANKS AND STANDARDS

	1/17/89	1/19/81/23/89
100 PPM STD	102	109 104
WT BLK 1	0.18	0.18 0.04
WT BLK 2	--	0.29 0

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

-- Samples lost

Detection Limit = 0.12 ug (coul)
Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K11

TYPE	AREA	DAY	MICROGRAMS		PPM	PPM
			A	B	HRS C	D
CITRATE AREA	1	TO 1	--	<1.0		
	1	TO 1	0.29	<1.0		
	1	TO 3	0.45	<1.0		
	1	TO 3	0.45	<1.0		
	1	TO 5	0.5	* 0.7		
	1	TO 5	0.3	* 0.5		
	2	TO 1	0.07	<1.0		
	2	TO 1	0.14	<1.0		
	2	TO 3	0.23	<1.0		
	2	TO 3	0.45	<1.0		
	2	TO 5	0.21	* 0.21		
	2	TO 5	0.14	* 0.14		
CITRATE BLANK	1	TO 1	0.07	<1.0		
	1	TO 3	0.3	<1.0		
	1	TO 5	<0.04	* <0.04		
	2	TO 1	0.07	<1.0		
	2	TO 3	0.5	<1.0		
	2	TO 5	<0.04	* <0.04		
IMPINGER AREA	1	1				<0.05
	2	1				<0.05
	1	2				<0.05
	2	2				<0.05
	1	3				<0.05
	2	3				<0.05
	1	4				<0.05
	2	4				<0.05
	1	5				<0.05
	2	5				<0.05

BLANKS AND STANDARDS

	2/14/89	2/16/89	2/20/89
100 PPM STD	100	104	103
WT BLK 1	0.04	0.07	0.04
WT BLK 2	0.00	0.09	0.04

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

* Analyzed by the coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant Limit = 0.4 ug (coul)

DATA FROM FIELD TEST K12

TYPE	AREA	DAY	MICROGRAMS		PPM	PPM	COMMENTS
			A	B	HRS	D	
CITRATE AREA	1	TO 1	<0.03	<1.0			
	1	TO 1	<0.03	<1.0			
	1	TO 3	<0.03	---			
	1	TO 3	<0.03	<1.0			
	1	TO* 7	<0.03	<1.0			
	1	TO* 7	0.25	<1.0			
	2	TO 1	<0.03	---			
	2	TO 1	0.1	<1.0			
	2	TO 3	<0.03	---			
	2	TO 3	0.03	<1.0			
	2	TO* 7	0.6	<1.0			
	2	TO* 7	0.5	<1.0			
CITRATE BLANK	1	TO 1	0.05	<1.0			
	1	TO 3	0.1	---			
	1	TO* 7	<0.03	<1.0			
	2	TO 1	<0.03	<1.0			
	2	TO 3	<0.03	<1.0			
	2	TO* 7	<0.03	<1.0			
IMPINGER AREA	1	1				<0.02	
	2	1				<0.02	
	1	2				<0.02	
	2	2				<0.02	
	1	3				<0.02	
	2	3				<0.02	
	1	4				<0.02	
	2	4				<0.02	
	1	7				<0.02	
	2	7				<0.02	

BLANKS AND STANDARDS

	2/22/89	2/24/89	2/28/89
100 PPM STD	99.6	98.5	96.7
WT BLK 1	0.07	0.0	0.0
WT BLK 2	0.14	0.0	0.0

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

--- Suspended matter, no PMA data.

* Samples sealed over weekend, total exposure only 5 days.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K13

TYPE	AREA	DAY	MICROGRAMS		PPM	COMMENTS
			A	B	HRS C	
CITRATE AREA	1	TO 1	0.07	<1.0		
	1	TO 1	<0.04	<1.0		
	1	TO 3	<0.04	<1.0		
	1	TO 3	<0.04	<1.0		
	1	TO 5	<0.04	<1.0		
	1	TO 5	<0.04	<1.0		
	2	TO 1	0.14	<1.0		
	2	TO 1	<0.04	<1.0		
	2	TO 3	<0.04	<1.0		
	2	TO 3	<0.04	<1.0		
	2	TO 5	<0.04	<1.0		
	2	TO 5	0.04	<1.0		
CITRATE BLANK	1	TO 1	<0.04	<1.0		
	1	TO 1	<0.04	1.6		
	1	TO 3	0.04	<1.0		
	2	TO 3	0.07	<1.0		
	2	TO 5	<0.04	<1.0		
	2	TO 5	0.04	<1.0		
IMPINGER AREA	1	1			<0.05	
	2	1			<0.05	
	1	2			0.09	
	2	2			0.09	
	1	3			<0.05	
	2	3			<0.05	
	1	4			0.06	
	2	4			<0.05	
	1	5			<0.05	
	2	5			<0.05	

BLANKS AND STANDARDS

	3/28/89	3/30/89	4/3/89
100 PPM STD	107	104	107
WT BLK 1	0.00	0.00	0.21
WT BLK 2	0.00	0.14	0.21

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

* Samples analyzed by coulometry.

**Samples were damp.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K13A

TYPE	AREA	DAY	MICROGRAMS		PPM	
			A	B	HRS C	PPM D
CITRATE	AREA	1	TO 1	21.77	*26.62	
		1	TO 1	16.52	* 17.55	
		1	TO 3	15.61	* 23.19	
		1	TO 3	23.54	* 22.65	
		1	TO 5	**>28	**,>28	
		1	TO 5	**24.5		
CITRATE	BLANK	1	TO 1	<0.04		
		1	TO 3	<0.04		
		1	TO 5	**<0.04		

BLANKS AND STANDARDS

	3/28/89	3/30/89	4/3/89
100 PPM STD	107	104	107
WT BLK 1	0.00	0.00	0.21
WT BLK 2	0.00	0.14	0.21

AVERAGE OF WT BLK 1 AND 2 USED TO CORRECT DATA

* Samples analyzed by coulometry.

**Samples were damp.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K14

TYPE	AREA	DAY	MICROGRAMS		BADGE HOUSING	
			A	B	BB = BLACK	WB = WHITE
CITRATE AREA	1	TO 1	>14.2	<1	BB	ROOF, HORIZ
	1	TO 3	>15.9	<1	BB	"
	1	TO 5	>14.6	<1	BB	"
	2	TO 1	2.4	<1	BB	ROOF, VERT., SOUTH
	2	TO 3	3.5	<1	BB	"
	2	TO 5	9.0	<1	BB	"
	3	TO 1	13.5	7.4	WB	ANTENNA, HORIZ
	3	TO 3	>16.3	>10.0	WB	"
	3	TO 5	>14.9	>10.0	WB	"
	4	TO 1	2.9	<1.0	BB	ANTENNA, VERT., SOUTH
	4	TO 3	5.0	<1.0	BB	"
	4	TO 5	>14.4	<1.0	BB	"
CITRATE BLANK	1	TO 1	<0.04		BB	BLANKS
	2	TO 1		<1.0	BB	
	1	TO 3	<0.04		BB	
	2	TO 3		<1.0	BB	
	1	TO 5	*<0.04		BB	
	2	TO 5	--		BB	
	3	TO 1	14.3		WB	
	4	TO 1		<1.0	WB	
	3	TO 3	>17.7		WB	
	4	TO 3		<1.0	WB	
	3	TO 5	*>25.5		WB	
	4	TO 5		<1.0	WB	
BLANK	OFFICE	TO 5	<0.04	<1.0	BB	
	OFFICE	TO 5	4.2	<1.0	WB	

*Problem with coulometric instrument, data may be suspect.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K15

TYPE	AREA	DAY	MICROGRAMS		PPM		COMMENTS
			A	B	HRS C	D	
CITRATE AREA	1	TO 1	2.7	<1			W
	1	TO 1	<0.04				B
	1	TO 1	0.05				B
	1	TO 3	0.79	*0.09			W
	1	TO 3	<0.04				B
	1	TO 3	<0.04	<1			B
	1	TO 5	0.29	*0.63			W
	1	TO 5	0.07	<1			B
	1	TO 5	<0.04	<1			B
	2	TO 1	1.93	<1			W
CITRATE AREA	2	TO 1	<0.04				B
	2	TO 1	0.05				B
	2	TO 3	0.36	*0.2			W
	2	TO 3	<0.04				B
	2	TO 3	<0.04				B
	2	TO 5	0.57	*0.84			W
	2	TO 5	0.07	<1			B
	2	TO 5	<0.04	<1			B
CITRATE BLANKS	1	TO 1	2.36	<1			W
	1	TO 1	<.04				B
	1	TO 3	.5				W
	1	TO 3	<.04				B
	1	TO 5	.41	*0.52			W
	1	TO 5	.14	*<0.04			B
CITRATE BLANKS	2	TO 1	.93	<1			W
	2	TO 1	<.04				B
	2	TO 3	.36	*0.17			W
	2	TO 3	<.04				B
	2	TO 5	.79	*0.7			W
	2	TO 5	<.04	<1			B
COLOR AREA	1	1			NC		
	2	1			NC		
	1	2			NC		
	2	2			NC		
	1	3			NC		
	2	3			NC		
	1	4			NC		
	2	4			NC		
	1	5			NC		
IMPINGER AREA	2	5			NC		
	1	1				<0.1	
	2	1				<0.1	
	1	2				<0.1	
	2	2				<0.1	
	1	3				<0.1	
	2	3				<0.1	
	1	4				<0.1	
	2	4				<0.1	
	1	5				LOST	
	2	5				LOST	

* Analyzed by the coulometric spike procedure.

Detection Limit = 0.12 ug (coul) Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K16

TYPE	AREA	DAY	MICROGRAMS		BADGE HOUSING
			A	B	
CITRATE PERSONNEL	1	1	13.66	<1.0	W
	1	1	<0.04	<1.0	B
	2	1	>16.8	1.0	W
	2	1	1.0	<1.0	B
	1	2	4.65	1.7	W
	1	2	0.14	*<0.04	B
	2	2	>14.3	<1.0	W
	2	2	<0.04	<1.0	B
	2	3	>14.3	2.7	W
	2	3	0.21	*0.07	B
	1	3	5.36	<1.0	W
	1	3	0.14	*<0.04	B

* Analyzed by the coulometric spike procedure.

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K17

TYPE	AREA	DAY	MICROGRAMS		PPM		BADGE HOUSING
			A	B	HRS C	PPM D	
CITRATE	AREA	1	TO 1	1.79	<1.0		W
		1	TO 1	<0.04			B
		1	TO 1	<0.04	* <0.04		B
		1	TO 3	4.6	<1.0		W
		1	TO 3	0.07			B
		1	TO 3	<0.04	<1.0		B
		1	TO 5	2.86	<1.0		W
		1	TO 5	<0.04			B
		1	TO 5	<0.04			B
		2	TO 1	1.39	<1.0		W
		2	TO 1	<0.04			B
		2	TO 1	<0.04			B
		2	TO 3	0.57	<1.0		W
		2	TO 3	<0.04	* <0.04		B
		2	TO 3	<0.04	* <0.04		B
		2	TO 5	<0.04	<1.0		W
		2	TO 5	<0.04			B
		2	TO 5	<0.04			B
CITRATE	BLANKS	1	TO 1	1.79	<1.0		W
		1	TO 1	<0.04			B
		1	TO 3	3.79	<1.0		W
		1	TO 3	<0.04			B
		1	TO 5	2.79	<1.0		W
		1	TO 5	<0.04			B
		2	TO 1	1.43	<1.0		W
		2	TO 1	<0.04	* <0.04		B
		2	TO 3	0.14	<1.0		W
		2	TO 3	<0.04			B
		2	TO 5	0.18	<1.0		W
		2	TO 5	<0.04			B
CITRATE	PERSONNEL	1		2.15	<1.0		W
		1		<0.04			B
		1		1.43	<1.0		W
		1		<0.04			B
		3		<0.04			B
		3			<1.0		W
		3		2.36			W
		5		<0.04	<1.0		B
		5		<0.04	<1.0		W
COLOR	AREA	1	1		** NC/VAN		SLIGHT YELLOW/PDAB
		1	1		"		"
		2	1		"		"
		2	1		"		"
		1	2		"		"
		1	2		"		"
		2	2		"		"
		2	2		"		"
		1	3		"		"
		1	3		"		"
		2	3		"		"
		2	3		"		"

DATA FROM FIELD TEST K17

TYPE	AREA	DAY	MICROGRAMS		PPM		BADGE HOUSING
			A	B	HRS C	PPM D	
IMPINGER AREA	1	1				<0.1	
	2	1				<0.1	
	1	2				<0.1	
	2	2				<0.1	
	1	3				<0.1	
	2	3				<0.1	
	1	4				<0.1	
	2	4				<0.1	
	1	5			--		LOST
	2	5			--		LOST

Badge housing: W = white, B = Black

* Samples analyzed by the coulometric spike procedure.

** GMD Dosimeter used as the color badge.
It contained two indicator sections:
Vanillin and P-Dimethylaminobenzaldehyde.

Detection Limit = 0.12 ug (coul)
Quant limit = 0.4 ug (coul)

DATA FROM FIELD TEST K18

TYPE	AREA	DAY	MICROGRAMS		PPM		BADGE HOUSING
			A	B	HRS C	PPM D	
CITRATE	AREA	1	1	<0.04	<1.0		W
		1	1	<0.04	*<0.04		B
		1	2	<0.04			B
		1	2	0.64	<1.0		W
		1	3	0.68			W
		1	3	<0.04	*0.05		B
CITRATE	PERSONNEL	2	1	1.5	<1.0		W
		2	1	<0.04	*<0.04		B
		2	2	3.9	<1.0		W
		2	2	<0.04	*<0.04		B
		2	3	2.5			W
		2	3	<0.04	<0.04		B
CITRATE	BLANK	1	1	0.5	<1.0		W
		1	1	<0.04	*<0.04		B
		1	2	0.5	<1.0		W
		1	2	<0.04	<1.0		B
		1	3	0.82			W
		1	3	<0.04	*0.05		B
COLOR	AREA	1	1			** NC/VAN, PDAB	
		1	1			** NC/VAN, PDAB	
		1	2			** NC/VAN, SLIGHT YELLOW/PDAB	
		1	2			** NC/VAN, SLIGHT YELLOW/PDAB	
		1	3			** /VAN, SLIGHT YELLOW/PDAB	
		1	3			** NC/VAN, SLIGHT YELLOW/PDAB	
COLOR	PERSONNEL	2	1			** NC/VAN, PDAB	
		2	1			** NC/VAN, PDAB	
		2	2			** NC/VAN, SLIGHT YELLOW/PDAB	
		2	2			** NC/VAN, SLIGHT YELLOW/PDAB	
		2	3			** NC/VAN, SLIGHT YELLOW/PDAB	
		2	3			** NC/VAN, SLIGHT YELLOW/PDAB	

Detection Limit = 0.12 ug (coul)

Quant limit = 0.4 ug (coul)

* Analyzed by coulometric spike procedure

** GMD Dosimeter used as the color badge.

It contained two indicator sections:

Vanillin and P-Dimethylaminobenzaldehyde

APPENDIX D

Facility data sheets from the field testing of the citric acid sampler. The information was collected by the industrial hygienist at the initiation of a test. It describes the area, operations, and chemicals in the test location.

FACILITY DATA SHEET

FACILITY NAME: WILTECH K7-516FACILITY POINT OF CONTACT: ARDELL THURLOWDATE: 11/14/87

DESCRIPTION OF FACILITY/AREA: WILTECH Building K7-516 is a chemical analysis laboratory. This facility is located within the Propellants storage and service area of Kennedy Space Center. To the east of the facility is a deionized water plant and gaseous nitrogen loading station. To the west is a hazardous waste facility and meter station. K7-516 is a 32,000 sq ft. facility housing several different laboratories. The facility includes a chemical analysis lab, vapor detection lab, fuel and oxidizer labs, a component calibration shop, a recharger maintenance shop and a vacuum pump/systems repair shop. mobile tankers and gas trailers are also present in the area parking lot.

DESCRIPTION OF OPERATIONS:

This facility is an industrial laboratory utilized by Kennedy Space Center to analyse any operations needed. This may include fuel and oxidizer purity level tests, precision cleaning of launch equipment, calibration of propellant detectors, analysis of potential hazardous waste and inspection of launch equipment.

CHEMICAL SUBSTANCES USED/STORED:

The facility is known to house at least 411 different chemical substances or reagents. Fuel and oxidizer are present in the building. All of these chemicals are used under a hood/ventilation system and the utmost care is taken when handling them. Technicians commonly use demineralized water, CCl₄, Isopropyl Alcohol, Methanol, weak acids, Freon 21, and numerous solvents.

FACILITY DATA SHEET

FACILITY NAME: HMF, m7-961FACILITY POINT OF CONTACT: E J. JandaDATE: 11/30/87 - 12/4/87

DESCRIPTION OF FACILITY/AREA: The Hypergol Maintenance Facility m7-961 is isolated in the southeast corner of the Industrial Complex at Kennedy Space Center. The area is surrounded by woods on all but the south side. Approximately one half mile to the south is m7-1061 which is utilized as offices and a control room. Within m7-961 there are two identical cells containing both fuel and oxidizer lines. These cells or rooms can be opened to the outside by large pull-up doors. A Mechanical Shop, Equipment Shop, and Office areas are also located within the building.

DESCRIPTION OF OPERATIONS:

The HMF m7-961 is the main facility to check out, service, and repair flight hardware on the OMS Pods and forward RCS before being sent to the OPF to be installed on an orbiter. The fuel and oxidizer found within the facility is flight residual and not stored or maintained in the building.

CHEMICAL SUBSTANCES USED/STORED:

The HMF m7-961 has both fuel and oxidizer lines in the east and west cells. This is flight residual and not stored or maintained in the building. Technicians may utilize Freon, methyl Ethyl Ketone, Isopropyl Alcohol, and 1,1,1 Trichloroethane to clean and maintain the flight hardware. Gaseous Nitrogen and Helium are also found within the facility.

FACILITY DATA SHEET

FACILITY NAME: m+o Paint Shop m6-486FACILITY POINT OF CONTACT: A. FranciscoDATE: 12/14/87 - 12/18/87

DESCRIPTION OF FACILITY / AREA:

The m+o Facility M6-486 is located in the Industrial Complex of Kennedy Space Center. The Industrial paint shop is located in the east corner of the M+O Facility. In addition to the paint shop the facility contains a carpentry shop, AC shop, electrical shop, tool crib, moving and roofing crew, machine shop, sheet metal and weld shop. No interior walls separate the individual shops. Vehicular traffic occurs adjacent to this facility and heavy mobile equipment is stored directly to the south.

DESCRIPTION OF OPERATIONS:

The m+o paint shop m6-486. is utilized by Kennedy Space Center for spray paint and sandblast operations. Silkscreens and stencils are also prepared in the shop. In addition paint equipment repair and maintenance also occurs in the facility.

CHEMICAL SUBSTANCES USED/STORED:

Technicians in the paint shop most often utilize methyl ethyl Ketone, lacquer thinner, xylene, mineral spirits, polyurethane thinner, epoxy thinner, zinc thinner, enamels, zinc primer, polyurethane varnish, coal tar epoxy paint, epoxy, lacquers and wash primers. A paint crib is also located in the shop to store the various paints and chemicals, and when technicians use the paints they wear MSA Full face respirators.

FACILITY DATA SHEET

FACILITY NAME: Hangar S Life Support South AnnexFACILITY POINT OF CONTACT: B. MartinDATE: 11/22-1/15/88

DESCRIPTION OF FACILITY/AREA: Hangar S South Annex - EG & G Life Support Building 1726 is located in the Cape Canaveral Air Force Station Industrial Area. The 52,000 sq.ft. facility is utilized for portable breathing air maintenance and Automated Payload Assembly and checkout. Behind the hangar is a hazardous waste staging facility, to the south is Hangar AF or SRO recovery Bldg. and to the northeast is building AE or missile assembly building. The south annex or building to the left of the facility's main hangar is utilized as a life support building. This is the facility we utilized for the study.

DESCRIPTION OF OPERATIONS:

Hangar S South Annex is utilized for checkout, maintenance, and repair of portable breathing units, SCAPE suits, helmets, gloves and boots. They also provide life support for individuals during hazardous operations, and are responsible for the upkeep of tube bank breathing lines and emergency egress breathing units or ELSA's for all Kennedy Space Center and Cape Canaveral Air Force Station.

CHEMICAL SUBSTANCES USED/STORED:

Technicians at the facility commonly use a Toluene/Freon mix, Freon 113, Isopropyl Alcohol, Seal Grip, and O-ring lubrication. These chemicals are utilized to clean, repair, and maintain the scape suits, gloves and boots.

606

FACILITY DATA SHEET
<p>FACILITY NAME: <u>Fuel Storage Area #1</u></p> <p>FACILITY POINT OF CONTACT: <u>Jerry Norman</u></p> <p>DATE: <u>2/9/88 - 2/12/88</u></p>
<p>DESCRIPTION OF FACILITY / AREA: Fuel Storage Area #1 is located at the Cape Canaveral Air Force Station. This Area is utilized for liquid propellant and fuel storage. There is a fuel, oxidizer, sodium hydroxide, and incinerator area, as well as a storage area for A-50, JP-5 and RP-1. To the north of the facility is the solid fuel storage area #2, and a non-destruct test laboratory. To the west of the area is the banana river and to the south is a new fuel storage area under construction, at this time.</p>
<p>DESCRIPTION OF OPERATIONS:</p> <p>Liquid Fuel Storage Area #1 is used to store and maintain various hazardous liquid fuels and propellants. These may include hydrazine, MMH, N_2O_4, NaOH, A-50, JP-5, and RP-1. These propellants and oxidizers are utilized for various launch programs and must be kept stable while they wait for future use.</p>
<p>CHEMICAL SUBSTANCES USED/STORED:</p> <p>Liquid Fuel Storage Area #1 stores liquid propellants, fuels and oxidizers to include N_2H_4, MMH, N_2O_4, NaOH, A-50, JP-5, and RP-1, various gaseous tanks and trailers of GN_2 and Helium are also present in the area.</p>

K07

FACILITY DATA SHEET

FACILITY NAME: Att Skirt Test Facility (ASTF)FACILITY POINT OF CONTACT: John Roberts III or Craig MastersDATE: 5/2-5/6/88

DESCRIPTION OF FACILITY / AREA: The Att Skirt Test Facility is located southeast of the Booster Assembly Refurbishment Facility. It consists of two levels, a fuel farm to the north, 2 test cells located to the east and west, a technical work shop, service and control room, and a scope suit-up area. To the south and east of the facility is swamp and to the north of the facility is the Lockheed Logistics equipment.

DESCRIPTION OF OPERATIONS: The area is utilized for acceptance testing and checkout of the SRB attskirt's thrust vector control system (tvc system). Hydrazine testing, fuel system testing, and loading and checking of the fuel system with nitrogen is performed at this facility. The nitrogen system is piped underground which is used for purging the fuel load.

CHEMICAL SUBSTANCES USED/STORED: The fuel storage area maintains hydrazine along with temporary drums of hydraulic fluid. Also, isopropyl alcohol is utilized for flushing G.O's in the fuel storage area.

FACILITY DATA SHEET

K08

FACILITY NAME: Aft Skirt Test Facility (ASTF)

FACILITY POINT OF CONTACT: John Roberts III or Craig Masters

DATE: 5/16-5/20/88

DESCRIPTION OF FACILITY / AREA: The Aft Skirt Test facility is located southeast of the Booster Assembly Refurbishment Facility. It consists of two levels, a fuel farm to the north, 2 test cells located to the east and west, a technical work shop, service and control room, and a scape suit-up area. To the south and east of the facility is swamp and to the north of the facility is the Lockheed Logistics equipment.

DESCRIPTION OF OPERATIONS: The area is utilized for acceptance testing and checkout of the SRB aftskirt's thrust vector control system (trc system). Hydrazine testing, fuel system testing, and loading and checking of the fuel system with nitrogen is performed at this facility. The nitrogen system is piped underground which is used for purging the fuel load.

CHEMICAL SUBSTANCES USED/STORED: The fuel storage area maintains hydrazine along with temporary drums of hydraulic fluid. Also, isopropyl alcohol is utilized for flushing QO's in the fuel storage area.

FACILITY DATA SHEET

1609

FACILITY NAME Rotating Service Structure Launch Complex 39B

FACILITY POINT OF CONTACT _____

DATE: 6/15-6/17

DESCRIPTION OF FACILITY: AREA The Rotating Service Structure (RSS) is located in Launch Complex 39B. The RSS supports the payload change out room, the FRCs room and propellant loading platforms. The badges were placed on the 107 foot level next to the MMH propellant loading lines.

DESCRIPTION OF OPERATIONS:

No operations were performed at the time of this test. During propellant loading operations, MMH is piped into the OMS pod thru the propellant lines located on the 107 foot level.

CHEMICAL SUBSTANCES USED/STORED:

None

FACILITY DATA SHEET

K10

FACILITY NAME KSC Beachhouse

FACILITY POINT OF CONTACT _____

DATE 7/11 - 7/15/88

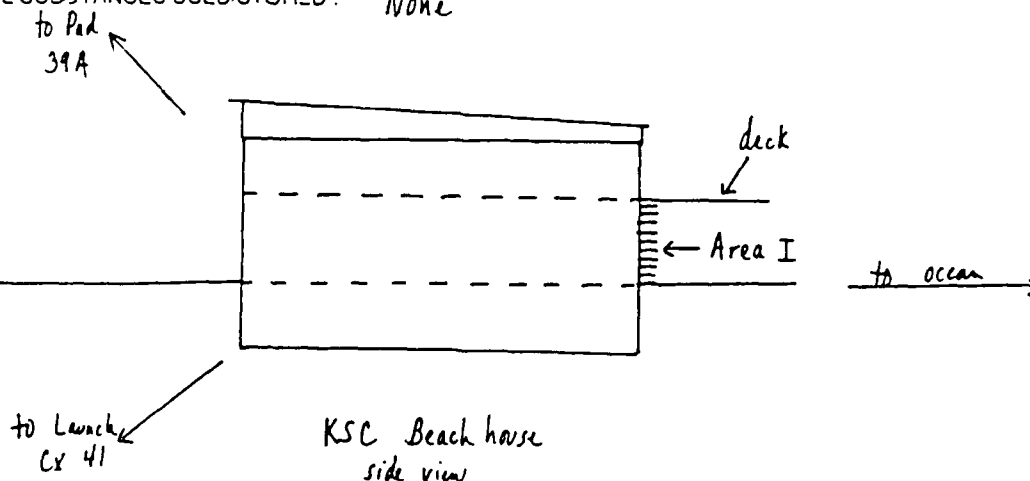
DESCRIPTION OF FACILITY AREA:

The KSC Beachhouse is located southeast of Pad 39A, northeast of Launch Complex 41, and approximately 70 yards from the ocean. It consists of 3 levels including a basement. There are ~~to~~ no facilities to the immediate north, south, or west of the beachhouse.

DESCRIPTION OF OPERATIONS:

A roofing operation continued throughout the week which included the removal of a tar/rock material and the preliminary work for the installation of a new roof. There were contaminants (i.e. dust, roofing debris, windblown sand) in the area throughout the week.

CHEMICAL SUBSTANCES USED/STORED: None



FACILITY DATA SHEET

K10A

FACILITY NAME: Rotating Service Structure Launch Cx 39B

FACILITY POINT OF CONTACT: _____

DATE: 1/16/89 - 1/20/89

DESCRIPTION OF FACILITY / AREA :

The Rotating Service Structure (RSS) is located in Launch Complex 39B. The RSS supports the payload changeout room, the FRCs room and propellant loading platforms. The badges were placed on the 107 foot level next to the MMH propellant loading lines.

DESCRIPTION OF OPERATIONS :

No operations were performed at the time of this test.

During propellant loading operations, MMH is piped into the OMS pod through the propellant lines located on the 107 foot level.

CHEMICAL SUBSTANCES USED/STORED :

None

FACILITY DATA SHEET

K11

FACILITY NAME: VAB breakrooms 6B10 & 10B6

FACILITY POINT OF CONTACT: _____

DATE: 2/13-2/17/89

DESCRIPTION OF FACILITY / AREA :

Rooms 6B10 (Area I) and 10B6 (Area II) are the only designated smoking areas in Tower 'B'. Room 6B10 is located on the sixth floor with an area of 182 sq. ft. Room 10B6 is located on the tenth floor, level 115'-9", with an area of 217 sq. ft. Normal occupancy during an 8 hr. workday is approximately 3 people for both rooms. Various offices are located adjacent to both rooms and across the hallway from the rooms.

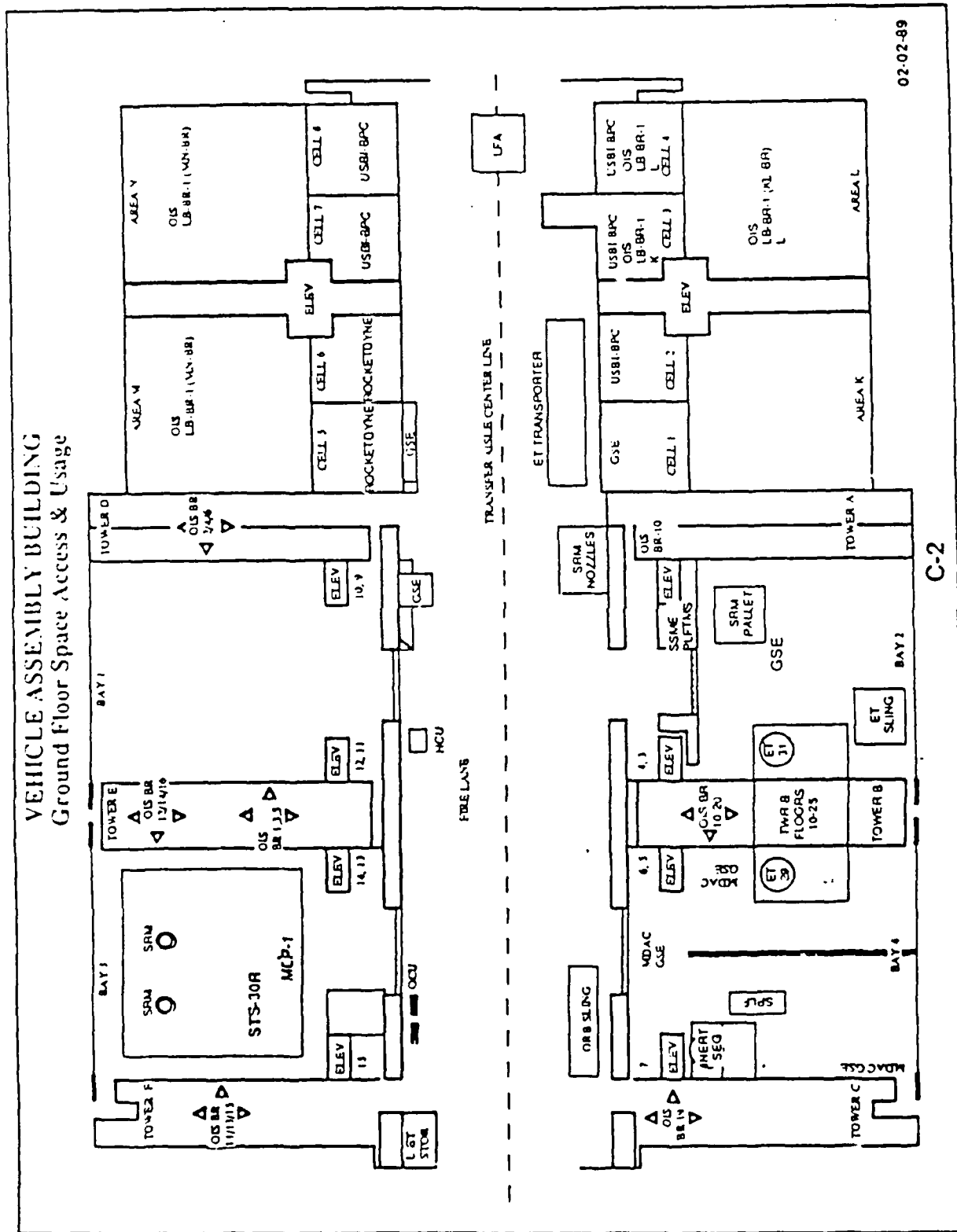
DESCRIPTION OF OPERATIONS :

The operations would include usual breakroom activities, such as smoking, eating, drinking, etc.

CHEMICAL SUBSTANCES USED/STORED :

None

VEHICLE ASSEMBLY BUILDING Ground Floor Space Access & Usage



FACILITY DATA SHEET

K12

FACILITY NAME: Aft Skirt Test Facility

FACILITY POINT OF CONTACT: Craig Martens

DATE: 2/21-2/27/89

DESCRIPTION OF FACILITY / AREA :

The Aft Skirt Test Facility is located southeast of the S&B Assembly Refurbishment Facility. It consists of two levels, a fuel farm to the north (Area I for this testing period), 2 test cells located to the east and west (test cell 1 which is located to the west is Area II for this testing period), a technical work shop, service and control room, and a scope set-up area. To the south and east of the facility is swamp and to the north of the facility is Lockheed Logistic equipment.

DESCRIPTION OF OPERATIONS :

The area is utilized for acceptance testing and checkout of the SRB aftskirt's thrust vector control system (tvc system). Hydrazine testing, fuel system testing, and loading and checking of the fuel system with nitrogen is performed at this facility. The nitrogen system is piped underground which is used for purging the fuel load.

CHEMICAL SUBSTANCES USED/STORED :

The fuel storage area maintains hydrazine along with temporary drums of hydraulic fluid. Also, isopropyl alcohol is utilized for flushing AD's in the fuel storage area.

FACILITY DATA SHEET

K13

FACILITY NAME: Orbiter Processing Facility H B 1

FACILITY POINT OF CONTACT: _____

DATE: 3/27-3/31/89

DESCRIPTION OF FACILITY / AREA :

The OPF is situated west of the VAB. It consists of two high bays and a low bay all in one large building, a 10,000 sq. ft. office and training annex, fuel and oxidizer deservicing pads, and GO_2 and CH_4 storage pads. The latter are well separated and placed well away from the main building. The hangar accommodates the Orbiter and the access platforms, which effectively surround the Orbiter for ease of access and maintenance. Area I was located on the 2nd level access platform to the right of the Orbiter nose cap without the FRCs installed. Area II was located on work level 10, panel 43 to the right of the GSE without the PHMS pad. Both areas were surrounded with various ground servicing and ground support equipment. (See attached for locations of areas)

DESCRIPTION OF OPERATIONS :

The OPF is used for processing returned Orbiters in preparation for reuse. Among the activities carried out in the OPF are Orbiter satting and deservicing, thermal protection subsystem refurbishment, payload removal and installation, and Orbiter active systems checkout.

CHEMICAL SUBSTANCES USED/STORED :

Chemical substances include ammonia, oxidizer, mono methyl hydrazine, tps chemicals and hydraulics.

K13

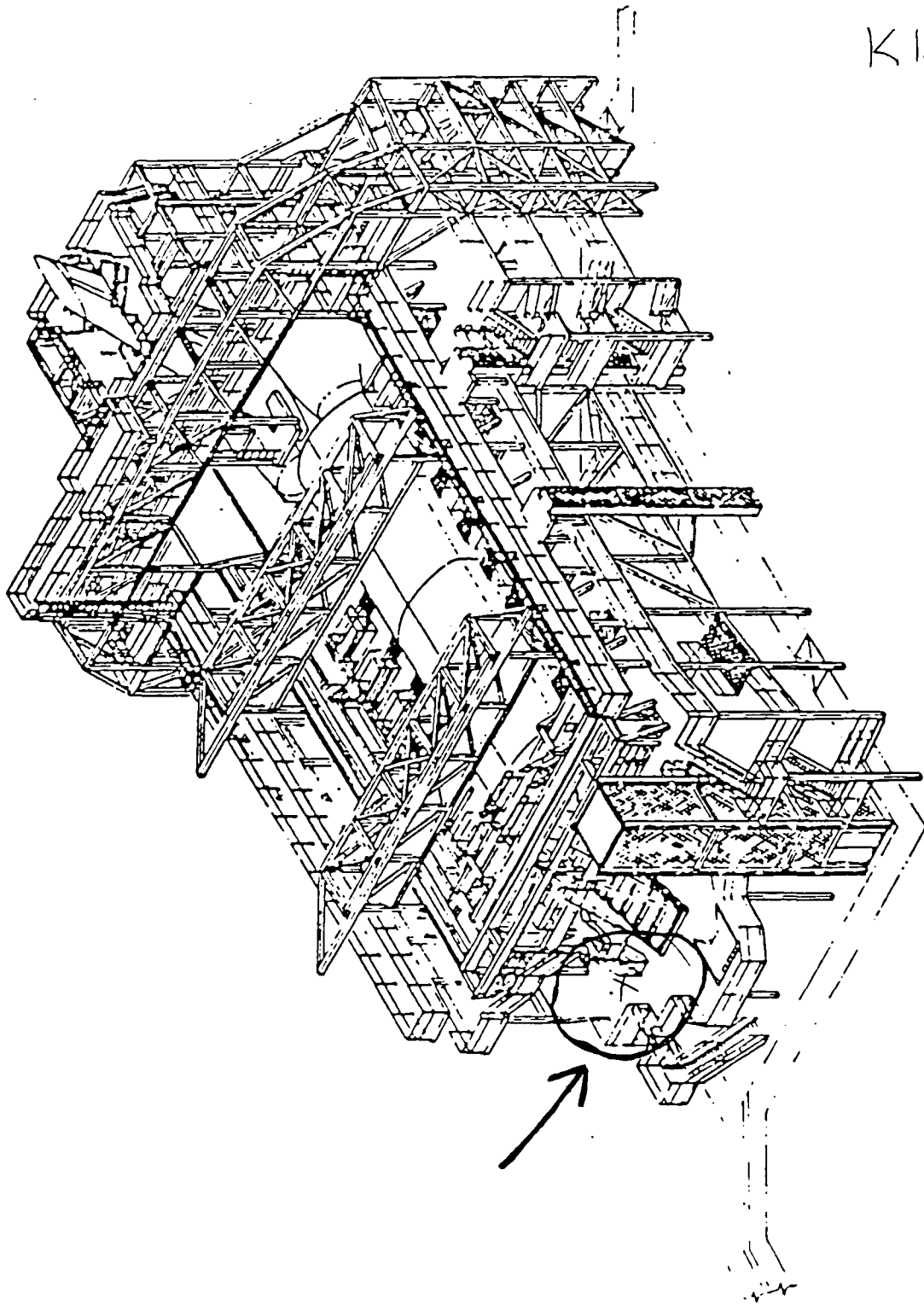


Figure 27-3. OPF Access Platforms
AREA I

FACILITY DATA SHEET

K13A

FACILITY NAME: Environmental Health Facility

FACILITY POINT OF CONTACT: _____

DATE: 3/27 - 3/31/89

DESCRIPTION OF FACILITY / AREA:

The Environmental Health Facility is located southeast of the ARF and northwest of Headquarters. It houses a health physics lab and an environmental sanitation and pollution control lab, in addition to a PLM lab located immediately behind the facility.

DESCRIPTION OF OPERATIONS:

This facility provides office space for approximately 50 Environmental Health employees and equipment for Industrial Hygiene, Environmental Sanitation and Pollution Control, and Health Physics. There was only one area for E and F badges which included the remaining badges after areas I and II at the OPF were set up. These badges were placed next to an antenna superstructure on the roof of this facility. The badges were facing directly toward the sun. No operations were performed on the roof during this week.

CHEMICAL SUBSTANCES USED/STORED:

None

FACILITY DATA SHEET

K14

FACILITY NAME: L7-1557 (Environmental Health Facility) Roof Top and L7-1557 East Antenna SiteFACILITY POINT OF CONTACT: S.W. BrownDATE: 5/9-5/12/89

DESCRIPTION OF FACILITY / AREA:

The Environmental Health Facility Roof Top is located southeast of the ARF and northwest of Headquarters. EHF houses a health physics lab and an environmental sanitation and pollution control lab. In addition, a PLM lab is located immediately behind this facility. The L7-1557 East Antenna Site was used as a remote monitoring site. It is approximately .3 mile east of the Environmental Health Facility, and is surrounded by woods.

DESCRIPTION OF OPERATIONS:

During this testing period, there were no operations in either of these areas. L7-1557 Roof Top was Areas I and II. Area I badges were placed flat and facing up. Area II badges were placed in a vertical position and were facing south. L7-1557 East Antenna Site was Areas III and II. Area III badges were placed flat and facing up. Area II badges were placed in a vertical position and were facing south. The badges in Area III were all white while the badges in the other areas were black.

CHEMICAL SUBSTANCES USED/STORED:

None

K14

KSC WEATHER CONDITIONS FOR 5/8-5/12/89

DAY	TEMPERATURE degrees F high,low	CLOUD COVERAGE OR VISIBILITY
Mon. 5/8	79,54	sunny
Tues. 5/9	82,61	sunny
Wed. 5/10	88,69	partly cloudy, heavy rains during the night
Thurs. 5/11	78,67	mostly sunny
Fri. 5/12	81,54	mostly sunny

TOTAL TIME EXPOSED BY DAY (MINUTES)

	Areas I and II	Areas III and IV
Mon. 5/8	328	333
Tues. 5/9	427	423
Wed. 5/10	377	356
Thurs. 5/11	464	469
Fri. 5/12	340	335

K15 & K16

Available Weather Conditions at KSC for 7/31-8/4/89

DAY	Temperature, degrees F high, low	cloud coverage/visibility
Mon. 7/31	data being used for a report, July unavailable	
Tues. 8/1	93, 74	mostly cloudy, 10 miles
Wed. 8/2	92, 74	mostly cloudy, 10
Thurs. 8/3	91, 73	mostly cloudy, 9
Fri. 8/4	91, 76	partly cloudy, 6

Test #16

FACILITY DATA SHEET

FACILITY NAME: m+o Paint Shop M6-486

FACILITY POINT OF CONTACT: A. Francisco

DATE: 8-2-89, 8-3-89, 8-4-89

DESCRIPTION OF FACILITY / AREA:

The m+o Facility M6-486 is located in the Industrial Complex of Kennedy Space Center. The Industrial paint shop is located in the east corner of the m+o Facility. In addition to the paint shop the facility contains a carpentry shop, AC shop, electrical shop, tool crib, moving and roofing crew, machine shop, sheet metal and weld shop. No interior walls separate the individual shops. Vehicular traffic occurs adjacent to this facility and heavy mobile equipment is stored directly to the south.

DESCRIPTION OF OPERATIONS:

The m+o paint shop M6-486 is utilized by Kennedy Space Center for spray paint and sandblast operations. Silkscreens and stencils are also prepared in the shop. In addition paint equipment repair and maintenance also occurs in the facility.

CHEMICAL SUBSTANCES USED/STORED:

Technicians in the paint shop most often utilize methyl ethyl Ketone, lacquer thinner, xylene, mineral spirits, polyurethane thinner, epoxy thinner, zinc thinner, enamels, zinc primer, polyurethane varnish, coal tar epoxy paint, epoxy, lacquers and wash primers. A paint crib is also located in the shop to store the various paints and chemicals, and when technicians use the paints they wear MSA Full face respirators.

Test #18

FACILITY DATA SHEET

FACILITY NAME: HMF M7-961

FACILITY POINT OF CONTACT: E.J. Janda

DATE: 8-9-89, 8-10-89, 8-11-89

DESCRIPTION OF FACILITY/AREA: The Hyperogol Maintenance Facility M7-961 is isolated in the southeast corner of the Industrial Complex at Kennedy Space Center. The area is surrounded by woods on all but the south side. Approximately one half mile to the south is M7-1061 which is utilized as offices and a control room. Within M7-961 there are two identical cells containing both fuel and oxidizer lines. These cells or rooms can be opened to the outside by large pull-up doors. A Mechanical Shop, Equipment Shop, and Office areas are also located within the building.

DESCRIPTION OF OPERATIONS:

The HMF M7-961 is the main facility to check out, service, and repair flight hardware on the OMS Pods and forward RCS before being sent to the OPF to be installed on an orbiter. The fuel and oxidizer found within the facility is flight residual and not stored or maintained in the building.

CHEMICAL SUBSTANCES USED/STORED:

The HMF M7-961 has both fuel and oxidizer lines in the east and west cells. This is flight residual and not stored or maintained in the building. Technicians may utilize Freon, methyl Ethyl Ketone, Isopropyl Alcohol, and 1,1,1 Trichloroethane to clean and maintain the flight hardware. Gaseous Nitrogen and Helium are also found within the facility.